GUIDELINES FOR THE USE OF WASTE CONCRETE FINES

by

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The production and maintenance of Portland cement concrete pavements creates a considerable amount of waste water, usually with a high pH and high levels of dissolved and suspended solids and the safe disposal of this material can be costly. The ability to reuse this waste water as mixing water into new concrete production would be a more cost efficient option, which would greatly reduce waste. Due to the presence of existing cement particles and elevated pH, waste water with both hydrated and unhydrated cement particles used as mixing water affects the performance of the concrete. These effects can potentially be beneficial, even if the water has a percent solids higher than recommended in current mix water specifications. Therefore, a method of quantifying the characteristics of the waste water is necessary to predict the performance of the concrete based on measurable properties of the waste water. This study quantifies the characteristics of the waste water, including pH, conductivity, and index of refraction. Models are then developed using a regression analysis. This is accomplished by characterizing waste water produced using multiple different sources of both grinding and wash out fines. Then, mortar properties are tested from mortar batches made with the characterized waste water, including compressive strength and set time. The laboratory data is then used for the development of regression equations for predicting the performance (set time and compressive strength) of the concrete, as a function of the waste water characteristics that are easily measured using in-line sensors. These relationships makes it possible to use waste water from a variety of sources in the production of new concrete while, being able to predict the effects of the inclusion

of the waste water on the concrete performance a priori. Finally, a mock set up of a plant water circulation system was constructed using in-line sensors for measuring the waste water properties. Concrete is then cast using water pulled from the lab-scale water circulation system to provide insight into the adequacy of the final models.

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1.0 INTRODUCTION

Concrete production and maintenance operations produce substantial amounts of waste water from grinding and wash out operations. Typical values acquired from pavement grinding maintenance activities are shown in Figure 1. This waste water has the potential to be reused in new concrete production as mix water, and experiments to date have been largely supportive of this form of recycling. Recycled waste water used in fresh concrete production must adhere to the same guidelines for any concrete mix water, ASTM C1602.

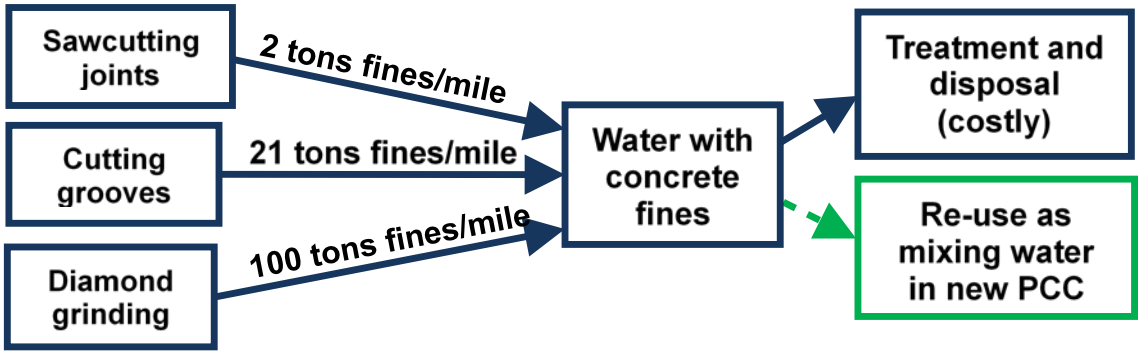


Figure 1. Typical production values for grinding fines maintenance activities.

Previous researchers have utilized a variety of material characterization methods and performance measurements, with set time and compressive strength testing being the most consistently measured indicators of hardened concrete performance, the waste water itself was most often characterized by its solids content. Previous work indicates a potential increase in early (3 or 7-days) compressive strength taken usually at. Explanations for this increase in early strength include improved particle packing and an expedited hydration rate. The wider range of

particle size distributions results from including the recycled water. The expedited hydration rate is attributed to the presence of hydrated cement particles in the waste water providing more nucleation sites, or to the presence of calcium hydroxide and elevated pH in the recycled water.

1.1 BACKGROUND

A substantial amount of waste water is produced at different stages throughout the lifespan of a concrete pavement, from standard production, maintenance, and removal practices. During production and following the placement of ready-mix concrete, concrete mixing trucks and equipment are washed out, a process which requires up to 318 liters of water per cubic meter of waste concrete (Geem et al, 1998). After hardening, construction procedures such as joint saw cutting and diamond grooving or grinding, as well as maintenance procedures such as diamond grinding or grooving, also require water to control dust and to cool the saw blades. Likewise, at the end of a concrete pavement's useful life, additional water for dust control is needed during crushing and removal operations. Each of these actions produce water with a high pH containing hydrated cement particles and possibly chemical additives, and requires proper disposal as it is categorized as an environmentally harmful material (Shogren et al 2009). These specific fines sources and their disposal requirements and effects on water are discussed below.

1.1.1 Grinding fines

The removal of hardened concrete, either when saw cutting joints, grinding or grooving produces a concrete dust, which must be controlled by water and results in a waste water product. Water is

also used during these construction procedures to cool the saw blades. Each removal practice produces a different quantity of fines but they are regulated and treated in a similar manner.

Saw cutting of joints occurs as soon as the concrete is sufficiently hard to support the saw cutting operation without generating spalling. Cutting joints in jointed concrete pavements produces approximately 2 tons of concrete fines per lane mile. Joint cutting occurs earliest in the lifespan of a concrete pavement relative to other grinding fines-producing maintenance activities. Therefore, while the concrete may be sufficiently hard for joint formation, it remains in a different stage of hydration than typical grinding and grooving projects. Waste water produced from joint saw cutting would therefore be expected to have a different chemical composition than those acquired from older concrete pavements.

Diamond grooving is performed to improve the skid resistance for aged pavements and newly paved pavement. This process is similar to that of diamond grinding but ultimately removes less concrete from the surface and, on average, produces only approximately 21 tons of concrete fines per lane mile. The material properties of the waste water from diamond grooving varies based on the age of the concrete. For maintenance operations on aged pavement, a lower pH is expected due to the carbonation of the surface. Diamond grooving completed on new construction is expected to contain both unhydrated and hydrated cement particles and produce a recycled water having a higher pH.

Diamond grinding is a ride restoration technique used for Portland cement concrete (PCC) slab surfaces where closely-spaced lines are cut into the concrete surface using diamond blades. It primarily removes a thin layer of the concrete pavement to decrease the roughness of the ride. This rehabilitation method removes joint and crack faulting, removes wheel path ruts from studded tires, corrects joint unevenness, and restores transverse drainage (AASHTO 1993). On

average, diamond grinding repairs reduce the slab thickness by 3/16 to ¼ in (Correa et al 2001). Typically, diamond grinding produces approximately 100 tons of concrete fines per lane mile. This dust is controlled by water to produce waste water containing hydrated cement particles as well as rock powder.

The effect of the fines on the water can vary widely and is a function of the specific concrete composition, the original quality of the wash water, and possible contaminants on the pavement surface during the maintenance operations. One of the most easily measured detrimental effects of this waste slurry is its high pH, where a value greater than 11.5 categorizes the water as an environmentally harmful substance (EPA Water Quality Act, 1987). In some cases, directly disposing this waste slurry onto the roadside soil was found to raise the pH from 6.3 to upwards of 9.4 (Shanmugam 2004). In a North Dakota study, the pH of concrete grinding slurry was found to fall between 11.6 and 12.5.

All three of these methods (diamond grinding, diamond grooving, and joint saw cutting) produce a waste water infused with hydrated cement particles that requires proper disposal. In the United States, regulations for the disposal of these waste materials is specified by each state, ranging from disposal below a roadway's shoulder to offsite disposal in containment ponds and landfills (DeSutter et al 2011).

1.1.2 Wash out fines

A second source of concrete fines occurs from washing out concrete trucks. The ready-mix concrete industry is responsible for large amounts of water consumption, specifically from concrete production, and washing out concrete mixing trucks and drums, required after each load (Tsimas et al 2011). The estimated daily requirement for wash water for each concrete mix truck

is 1500 L (400 gallons). This waste water contains hydrated cement particles and typically has an elevated pH, which requires it to be classified and treated as a hazardous material by both European and United States environmental regulations. Traditionally, this wash water has been disposed of in settling ponds at ready-mix concrete facilities to allow for the suspended solids to settle and the water to slowly filter. The settled suspended solids can then be dried and sent to a landfill. However, given the elevated pH of this product and recent categorization as a potentially hazardous material, requirements for disposal and treatment are becoming more stringent both within the United States and worldwide. The presence of dissolved calcium hydroxide leads to a high pH in the concrete wash water and the water has also been found to contain other dissolved solids, including sulfates, hydroxides, and chlorides, as well as traces of oil, and grease (Elchalakani et al 2012).

In the United States, the Environmental Protection Agency Water Quality Act, part 116, categorizes concrete wash out water as a hazardous substance based on the regulations of corrosivity and the high pH of the wash water (Chini. 1996). The Environmental Protection Agency published recommendations for the recycling of concrete wash out water suggest filtering the waste water through a series of filters and reusing the final water as wash out water for more concrete mixing trucks. Alternatively, the filtered wash water can be treated until its metal levels and pH fall within acceptable limits for standard disposal. The EPA also recommends recycling concrete aggregate if separation from the mortar matrix is feasible (EPA 1987).

In the United Kingdom, waste water has traditionally been disposed of in landfills, but recent regulations from the Environmental Agency have categorized water with a pH higher than 11.5 as hazardously alkaline and an additional tax for landfill disposal of concrete waste water

has been enforced (Sealey et al, 2001). The combination of the increasing cost of proper disposal and treatment with increasingly stringent disposal regulations has led to the reuse of concrete wash water in many countries.

1.2 RESEARCH OBJECTIVES

The primary objectives of this study are: 1) to develop and execute a laboratory study to robustly characterize recycled concrete waste water, 2) to develop a complete database through laboratory experiments in order to investigate the effect of the characterized recycled concrete water into fresh concrete production and measuring two primary performance parameters, 3) to investigate the relationship between the characterization of waste water and the performance of the concrete made using this water and develop prediction equations through regression analysis to describe this relationship and make valid predictions of expected behavior, 4) to create a full scale laboratory mock up to validate the constructed prediction equations, 5) to develop guidelines for industry personnel for using the prediction equations and investigate the model sensitivity.

The results of this study can be used by ready-mix concrete plants or other entities that must process concrete waste water. The results will make it possible to reuse stored concrete waste water in the production of new concrete; thus providing an economically viable alternative to costly water treatment.

1.3 RESEARCH APPROACH

To accomplish these research objectives, a laboratory testing program was designed to populate a rigorous and complete database to better understand the relationship between concrete waste water and the performance of mortar specimens. Collected samples of concrete waste water were first processed and characterized based on concentration and according to four different measurement parameters. These same samples of waste water fines were then used in the preparation and testing of mortar mixtures, which were tested both for set time and for 3-day and 28-day compressive strength testing. Then, specific relationships between the characterization of the waste water and the performance of the mortar testing specimens are established.

Finally, the prediction models developed are validated using a mock water recirculation set up with in-line sensors in order to validate the established model, developed using mortar data, against the concrete construction. Additionally, a set of guidelines is then developed for users in order to show sensitivities and recommended usage of the prediction models.

1.4 STRUCTURE OF THE THESIS

The structure of the thesis is as follows: Chapter 2 presents a literature review that includes a review of current regulations for concrete mix water. Previous work done to either characterize concrete waste water or to investigate the effect of using recycled fines or waste water in concrete is then reviewed and discussed. The possible effects of the inclusion of recycled fines on concrete, and specifically the effects on hydration and particle packing are investigated and discussed. Chapter 3 presents the methodology of the experimental investigation. The selection

of the recycled fines sources is discussed followed by a description of the preparation technique. The laboratory materials characterization procedure is then discussed, including a description of the equipment and the procedure. Finally, the mortar testing procedure and equipment, for both the set time testing and compressive strength testing are discussed.

Chapter 4 discusses the results from the experimental investigation, beginning with the materials characterization of the six types of recycled fines followed by the mortar testing. Chapter 5 then discusses the development of the predictive equations using the data and results presented in Chapter 4. Different methods of data processing, as well as the model development, are presented and the models are individually presented and discussed.

Chapter 6 then discusses the implementation of the model through a mock up water recirculation system. The developed predictive equations are then used for predictions and concrete specimens were cast to verify the accuracy and predictive capabilities of the developed model. Chapter 7 then presents user guidelines for the models developed, such that sensitivities are investigated and discussed and ranges of use are recommended. Finally, Chapter 8 presents conclusions of the research including limitations and suggestions for future work.

2.0 LITERATURE REVIEW

2.1 MIXING WATER REGULATIONS FOR NEW CONCRETE CONSTRUCTION

One primary obstacle in using recycled wash water for concrete mixing water are the governing standards for mixing water for the production of fresh concrete. Standard specifications exist for the quality and content (including total solids, chlorides, alkalis, and sulfates) of mixing water used for fresh concrete production in both the United States (ASTM C1602) and Europe (EN 1008). Both specifications outline expectations and requirements from all water sources and provide additional quality control guidelines specifically for the reuse of water recovered from the concrete industry, which includes wash out water as well as water from grinding and cutting operations.

Both sets of standards outline two absolute requirements for any mixing water used regarding both compressive strength and time of set. Any mixing water used must meet the requirement that the 7-day compressive strength is at least 90% of the mean compressive strength of the control mixture, which is made with distilled or deionized water. In the ASTM C1602 specification, the time of set must not vary from the control mixture by less than 60 minutes or more than 90 minutes. The EN 1008 requirement also specifies that the time of set cannot vary by less than 60 minutes of the control set time but also cannot vary in either acceleration or retardation by 25% of the control mixture set time. These regulations pertaining

to set time and compressive strength serve as the absolute minimum requirements for any water used for new concrete production.

The mass of solids is also governed by both specifications and is estimated in both through the water density. The ASTM C1602 specification lists an optional limit of total solids content of 50,000 ppm, to be specified by the purchaser or concrete mixture designer. Limits on the composition of these solids are also given with limiting values provided for chloride, sulfates, and alkalis content.

Limitations on the concentration of dissolved chloride ions are given primarily because of the possible corrosion of embedded reinforcing or prestressing steel. Variable limits are specified based on the use of the concrete and whether or not it will contain reinforcing steel. ASTM C1602 limits this value to 500 ppm for prestressed concrete and bridge decks and 1000 ppm for other reinforced concrete while the EN 1008 specification limits the chloride content to 500 mg/l for prestressed concrete, 1000 mg/l for reinforced concrete and 4500 mg/l for plain concrete.

Potentially expansive reactions and consequent deterioration by sulfate attack drives the limitations of sulfate content in mixing water. This reaction can be expedited or exacerbated from environmental conditions, such as high sulfate soils. As a result, the ASTM C1602 specification limits sulfate content to 3000, ppm while the EN 1008 specification limits sulfate content to 2000 mg/l.

Finally, alkalis such as Na_2O and K_2O must also be limited as high concentrations of alkalis have been found to reduce concrete strength while accelerating the hydration process (Kosmatka 2002). This ultimately lowers 28-day strength despite accelerating the early strength. Additionally, high alkaline water can instigate the development of alkali-silica reactions in the final concrete. The concentration of alkalis is limited to 600 ppm and 1500 mg/l for the ASTM

C1602 and EN 1008 specifications, respectively. The EN 1008 provides a leniency with this limit, however, and specifies that water with alkali content higher than this specification is acceptable for use if proactive measures are taken to prevent alkali-silica reactions.

Additionally, EN 1008 provides limitations for miscellaneous other contaminants that could possibly be found in all mixing water and considered harmful, such as sugar, phosphate, nitrates, lead and zinc. Restrictions are also outlined in EN 1008 for non-harmful contaminants including oils, fats, detergents, color, suspended matter, odor, pH, and humic matter. It should be noted that only a lower limit of a pH of 4 is given for mixing water in this specification and no upper limit is specified.

The ASTM requirement offers no further requirements exclusively for the reuse of water except to suggest the use of hydration stabilizing admixtures (HSA) for water with a density greater than 1.05 g/L. This is in order to meet the two primary base requirements of mixing water: compressive strength and set time. Hydration stabilizing admixtures can reduce the rate of hydration of cement by a pre-determined amount (based on dosage) to manipulate the time of set and are frequently used for extending the time frame of concrete delivery.

In addition to the requirements outlined for all mixing water, supplementary specifications are given in the EN 1008 specification, particularly for the use of recycled concrete water as a replacement of fresh mix water. The specification assumes that no adjustments are made in the concrete mix design. Based on this, a limit on these additional solids is given as less than 1% of the total mass of the aggregates in the concrete mix. Any unique requirements beyond those for standard concrete, such as architectural concrete, prestressed concrete, air entrained concrete, or concrete in extreme climate conditions, must be additionally evaluated with respect to effects of

recycled concrete for that specific intended use. Additionally, the reuse of recycled water should be evenly distributed through concrete production over the course of a day.

The density specification is used to estimate total solids content in the recycled concrete mixing water. Water with a density greater than 1.01 kg/l (which would indicate a non-trivial amount of residual concrete fines in the water) requires agitation when used to maintain a homogeneous distribution of the solids. The mass of solid material in the water is a more flexible and discretionary quantity. Specifications state “for some production processes, a greater quantity of solid material may be used provided satisfactory performance in concrete can be demonstrated.” (DIN 1008)

The Portland Cement Association recommends total solids below 50,000 ppm, because concerns are raised with higher levels regarding the effects on set time, concrete efflorescence, possible rebar corrosion, volume instability, reduced durability and reduced workability of the final concrete product (Geem 1998). The ASTM standard, however, holds set time and compressive strength of the final concrete product as its primary concern.

2.2 CHARACTERIZATION OF RECYCLED WATER AND PERFORMANCE PARAMETERS

Given the broad spectrum of concentrations, materials, and degree of hydration of waste water as well as varying sources, material characterization of waste water is required to give some correlation to concrete performance. This can include, but is not limited to, testing pH, organic matter content and electric conductivity. Previous work has included measuring varying concrete properties, to compare with specifications. These hardened properties always include

compressive strength measurements. Although, both fresh and other hardened concrete properties have also been measured in an attempt to provide some indication of the waste water suitability for reuse, as will be further discussed below.

As per the specification requirements, characterization of the recycled water itself included measurements of soluble salt, chloride, and sulfate content as well as the total solids content to ensure compliance with the specification limits. Other measurements taken to characterize the mix water quality included measuring mineral, salt, and miscellaneous impurities contents (Borger et al 1994). Solids content was also used to estimate density and percentage of solids by mass based on loss on ignition measurements (Lobo et al 2001). Dissolved solids and conductivity were also measured to provide some indication of concrete performance and solids content of both hydrated and unhydrated cement particles (Ekolu et al 2010). Likewise, specific gravity can be measured and used through linear relationships to estimate total solids content of the slurry water (Chatveera et al 2009).

Properties used to measure the performance of fresh concrete included the slump test for workability. Workability is a primary concern for concrete made with recycled waste materials because of the expedited increase in set time of the mortar and concrete possibly due to the expedited hydration. Consistently, it was found that increasing the quantity of waste fines in concrete both shortened the set time and decreased the workability of the mix measured either through slump testing for concrete or flow measurements for mortar (Sandrolini et al, 2001).

Sandrolini et al's work (2001) focused primarily on the effect of the microstructure on concrete performance and therefore also included studying grain size distributions to quantify the fineness of the solid matter. Set time, which is also a constraint in mixture specifications, was also measured to indicate rate of hydration (Borger et al 1994). Sulfate resistance of the mortar

should also be measured if the mix water quantities have indicated that elevated sulfate levels are present.

Concrete durability can be improved by fine filler effects and a reduction of concrete capillary water absorption and porosity. Measuring the concrete's porosity has also provided some indication of performance due to its relationship to the grain size distribution and correlation to the mortar mix density (Sandrolini et al 2001). Similarly, an increase in the resistance of the concrete to sulfate attack, as measured through expansion mortar bar testing, provided an indication of the increased density of the mortar matrix (Borger et al 1994).

2.3 EFFECT OF USING RECYCLED FINES

Quantifying the effects of using recycled wash water and waste water has been largely unexplored. Most projects that have tested the effects of including recycled concrete waste fines water in the production of new concrete have not established a relationship between characteristics of the water and properties of the concrete produced. Rather, research to this point has included ensuring that recycled water falls within mixing water specifications and that the final concrete produced falls within concrete strength specifications. Considering the two primary concerns of concrete specifications being time of set and compressive strength, three trends were observed: 1) the inclusion of waste water increased short-term concrete and mortar strengths (3 or 7-day testing) and 2) the inclusion of waste water had a negligible effect on 28-day strength results and 3) the time of set was highly varied. These differences have been primarily attributed to particle packing effects and acceleration of the concrete hydration reaction, which will be discussed at the end of this section. Both particle packing effects and

acceleration of the hydration reaction have been thought to contribute to potential early strength gains but the amount that either contributes to compressive strength gain remains unknown (Jaturapitakkul, 2011).

Limited work has been completed so far using recycled water as mixing water for new concrete and testing procedures have been largely inconsistent, leading to inconsistent and incomparable results. Most have maintained water requirements of either or both ASTM C94 and EN 1008 standards. Different methods of characterizing fines and fine properties were used and in some cases, a hydration stabilizing admixture was used to widen the period of time when concrete fines could be used. Previous studies testing plain concrete made with recycled water will be discussed first followed by studies that included additives and admixtures in the concrete mix and test methodologies. The parameters measured, and results for each of these studies will be discussed.

2.3.1 Plain concrete testing

Work by Sandrolini et al (2001), which evaluated water against both ASTM and EN 1008 standards collected water at varying levels of settling. The water was characterized by pH, amounts of suspended matter, and evaporation residue following testing as outlined in EN 1008. The fineness of the solid matter was calculated by allowing the volume of solids to settle and calculating evaporation residue. Sample compositions were identified through measuring soluble salts, chlorides and sulfates before testing and by using laser grain size measuring equipment to outline grain size distributions and X-ray diffraction to gain insight into chemical composition. The total solids never exceeded the 50,000 ppm specified in ASTM C94.

Following the initial characterization, both mortar prisms and concrete cubes were cast and the workability and water absorption were also measured. The w/cm ratio was held constant and, as a result, the workability decreased as more recycled wash water was used. The compressive strengths revealed 7-day strengths were higher than the control and 28-day strengths were slightly lower than the control, but still within both sets of requirements for mix water. No relationship was detected between characteristics of the solids content of the wash water and the final compressive strength. The result from the mortar prisms, however, showed lower strengths than those for the control at 7-days but comparable or better 28-day strengths. The higher 28-day strength of the mortar samples, as opposed to the lower 28-day strength of the concrete, could indicate that the type of coarse aggregate (limestone) used, could have contributed to the observed increase in strength for the concrete.

Concrete made with recycled water also exhibited a lower porosity and water absorption, as estimated by the volume of water absorbed by a concrete sample submerged in water. This decrease in absorption was attributed to the fine suspended particles behaving as a filler, thus decreasing the effective pore size. This would be consistent with the lower porosity values as well.

Tsimas et al (2011) conducted similar testing but sought to more thoroughly investigate the composition of the recycled concrete water. Wash out water samples were collected and progressively diluted to obtain a wider spectrum of concentrations of concrete fines. All water samples fulfilled both specifications for the amount of total solids and all had pH levels over 11.5, thus categorizing them as hazardous materials. A high loss on ignition suggests that large amounts of calcite were present in the water, possibly from the fine fractions of the fine aggregate. An analysis of the solids content of the sludge water revealed the most common solid

to be CaO followed by SiO_2 and negligible amounts of all other solids. A mineralogical analysis of the water using X-ray diffraction revealed that the fines material was comprised of mostly calcite and silicon oxide as well as $Ca(OH)_2$. $Ca(OH)_2$, more commonly known as portlandite, is a product of cement hydration, implying that some of the cement particles in the recycled water were already hydrated. It was found that most 7- and 28-day strengths exhibited a slight improvement over the strength of the control mix. Due to the fineness of the particles in the recycled water, this slight strength gain was attributed to improvements in the packing index. The packing index is defined as the ratio of volumes of an individual particle and the unit cell. Contrary to other studies, no impact on workability of the concrete was found and the slump was affected only by the addition of admixtures. Also, no significant change to set time was observed.

Similar work performed by Su et al (2002) also focused on more thoroughly categorizing the properties of the recycled water and tested water with a variety of total solids concentrations and measured pH, turbidity, total solids, chloride ion content, and sulfate content. Wash water was taken from varying depths of sedimentation pools to obtain a wider spectrum of particle concentrations. All pH levels were found to exceed 11.0, and both turbidity and total solids were found to increase with increasing particle concentration. All measures of performance fell within the limits as outlined by the specifications. Chloride and sulfate levels fell within the ASTM C1602 and EN 1008 standards. The mortar time of set fell within -10 minutes and +30 minutes of the control mixture, well within the specification limits. Both the 7-day and 28-day compressive strengths were above the base requirement of 90% of the control strength. While 28-day compressive strengths fell below control values, but still within limit, the 7-day compressive strengths (early strength) exceeded the control strengths. Additionally, the measured

compressive strength of the concrete samples increased as the concentration of the solids in the water increased.

2.3.2 Concrete containing additives and admixtures

Given the increased set time and decreased workability sometimes found when using recycled water in concrete mixtures, researchers have evaluated chemical admixtures and cementitious replacement materials to counter some of these undesirable effects. Hydration stabilizing admixtures were used to counter the decreased hydration rate due to the inclusion of concrete waste water. Hydration stabilizing admixtures first stabilize the hydration reaction for a period of time (depending on dosage) and then activate hydration. This two-step process results in the slowing of cement hydration followed by a sudden acceleration of the hydration process.

Work performed by Ekolu et al (2010) included experimenting with mortars and concrete mixtures made with recycled concrete wash water and mixing samples with and without slag as a replacement for cementitious materials. Tests completed on the mortar and concrete included slump and flow, unit weight, set time, total heat of hydration, compressive strength, and permeability. The total dissolved solids of the recycled was within the EN 1008 specification and were approximately 20 times greater than the control mix water. Total dissolved solids, conductivity, and pH were measured for each water sample and chemical impurities were measured (chlorides and sulfates) to compare with the EN 1008 requirement. All recycled water used fell within the requirements for total solids, chlorides, and sulfates.

Set time decreased when recycled water was used but still fell within the limits outlined in EN 1008. When slag was used as a replacement for cementitious material, while the set time was higher than that for the control mixture. Slump was also found to steadily decrease with

increasing concentrations of solids in the wash water. Concrete strength, however, fluctuated, whereas the strength of the mortar did not, indicating a possible adverse reaction with the coarse aggregate. The mortar strength consistently increased when using recycled fines water with 28-day strengths exhibiting an 8% increase over the control. Overall, concrete prepared with the recycled concrete water showed a decrease in workability and an increase in unit weight, indicating a denser mortar matrix.

Research performed by Borger et al (1994) used stabilizing admixtures to control the rate of hydration. The compressive strength, set time, workability and sulfate resistance of mortars were investigated. Rather than keep a constantly agitated supply of waste water with a stabilizing admixture, this effect was approximated by controlling the time since the cement comes in contact with the water for the wash water. This time ranged from 2 hours to 48 hours. The stiffest mortar was produced using wash water between 2 and 4 hours old, likely due to the heightened reactivity of the cement particles at this point. The mortars became equally stiff to that of the control mixture after the wash water had aged 8 hours.

The greatest strength gain for the mortar mixtures was achieved when using the 2-hour old wash water and the lowest strength gain was obtained when the 24 and 48-hour old wash water was used. This was attributed to the reduction in the water-cementitious materials ratio (w/cm) due to the addition of the fines water. Since the cement content was not adjusted to account for the additional solids in the fines water, the addition of this water resulted in an increase in the total cement content. Without the inclusion of a stabilizing agent, the set time for the control mixtures varied up to 25% from the control mixture. The accelerating affects from the wash water were controllable with stabilizers. The 2-hour old water also best resisted sulfate attack. The age of the wash water had the greatest impact on the concrete strength. The 28-day strength

increased by 20% and an increase in strength was generally observed for ages of wash water 8 hours old or less. Overall, the expansion of the cement, as determined by mortar bar expansion testing, increased with the increasing age of the wash water. This was likely because the overall cement content of the mix increased as the wash water aged, thereby increasing mortar matrix density and reducing expansion.

Lobo et al (2001) simulated continuously agitated slurry tanks with a laboratory set up that included a motorized paddle. The solids content was varied with the time of day to better simulate truck wash out variability conditions. The total solids content varied between 25 and 40 percent solids by mass. Unlike other previous work, Lobo included a solids content up to four times more than the ASTM total solids limit. Density and percentage of solids by mass were measured as well as loss on ignition and insoluble residue.

Concrete samples were mixed to a target slump value rather than maintaining a constant w/cm ratio. Therefore, mixture compositions were more highly varied than in other previous work. Density, initial set time (using the Vicat test) and temperature were measured so the measured properties could be matched to a heat signature curve. The amount of mix water required to obtain the desired slump increased as the concentrations of solid particles in the slurry water increased. The increase in water demand was proportional to the amount of solids as well and the age of the slurry, with slurry water aged past one day requiring significantly higher amounts of water.

The initial set time of the control was 4.9 hours while the largest variation occurred for recycled water with the highest concentration of solids with a set time of 4 hours. This accelerated hydration rate was attributed to the hydrated cement and calcium hydroxide (hydrated lime) in the slurry. There was a noticeable reduction in 28-day concrete strength,

which is most likely due to the addition of extra water needed to obtain the 5 in slump required. The weakest concrete samples were also those which required the highest levels of additional water to achieve the desired slump and therefore also had the highest w/cm ratios. Younger slurries which were aged less than 4 hours, had higher strengths possibly due to the additional cement provided by the unhydrated cement particles. Mixtures with high water contents had higher levels of drying shrinkage and permeability, also likely due to the increased water content itself rather than the composition of the recycled water.

Following this first phase of testing, Lobo et al then included HSA into a second phase of testing. By controlling the HSA dosage, compressive strength results for the control mixture was similar to the HSA treated concrete made with 7-day old slurry water. Concrete made with the 7-day old slurry performed worst relative to the control mixture when HSA was not added. Therefore, while 7-day slurry water, and water outside of the ASTM/EN requirements was found to be the most detrimental to concrete performance when left untreated, treatment with HSA was able to rectify these effects. It should be noted that 4-hour old slurries did not require treatment with HSA to fall within these testing limits and most closely aligned with the performance of the control mixture.

A more comprehensive study by Chini et al (2000), included both standard (Type I) and bridge deck (Type II) concrete mixtures made with HSAs coupled with water reducing admixtures. Concrete mixtures were also made to check dosage effects on air-entraining and water reducing admixtures when hydration stabilized wash water was used. Type II concrete for bridge deck use was also prepared. For all testing, fly ash was used as a cementitious replacement material in the control mixture. The test concrete was mixed to a standard slump, and consequently had different w/cm ratios varying from 0.48 to 0.55. Three different limestone

coarse aggregates were used from three different local sources. Properties measured for each concrete mixture included temperature, slump, unit weight, air content, set time, compressive strength, flexural strength, drying shrinkage, resistance to chloride-ion penetration, and sulfate expansion. It was found that for concrete mixtures made with a chemical stabilizer for re-used wash water, the two primary differences were increased drying shrinkage and reduced set times.

Work by Elchalakani et al. sought to test the effects of using completely recycled concrete, that is, concrete made using both recycled concrete wash water as well as recycled concrete aggregates. The recycled water was obtained from the wet recycling process during concrete production. The mechanical properties of the finished concrete were found to be highly dependent on the quality of the recycled aggregate and water used. Most specimens tested fell within the quality standards described from the ASTM C1602 and EN 1008 standards. It was found that when slag was used as a replacement for the cementitious material that the strength and durability of the concrete increased. The highest strengths were achieved from fully recycled concrete (with 100% of both recycled aggregate and recycled water) with 80% slag replacement.

Chatveera et al. conducted a similar experiment but included concrete admixtures coupled with additives. The recycled water used did not satisfy ASTM C1602 because it contained a total solids content of 56,000 ppm, exceeding the 50,000 ppm limit imposed by ASTM C1602. A linear relationship between the total solids content and the specific gravity of the recycled water was obtained. The recycled water also contained a high pH and a high loss on ignition. The particle size distribution was measured for each concrete component and while the distributions were close, the overall average size distributions ranged from coarsest to finest for the recycled fines, fly ash, and Portland cement, respectively.

The concrete mixtures were mixed to obtain a specified slump measurement, therefore the w/cm varied. Samples having a total solids content of less than 5% had a compressive strength most comparable to that of the control. However, the set times increased substantially (by more than 90 minutes), This is greater than that allowed under ASTM C1602. Through experimentation, it was found that the optimal recycled water content that fell within the ASTM specification for strength and set time contained between 5.4 and 6.1% solids. It is interesting to note that this optimal percentage of total solids content for achieving ASTM specifications falls outside of the 50,000 ppm limit of total solids suggested by the ASTM C1602 specification.

The concrete made with recycled water without any additives or admixtures was ultimately found to have a longer set time than the plain control concrete. Concrete made with super plasticizers and recycled water was found to have a noticeably reduced set time and slump. Concrete made with recycled water had compressive strengths lower than plain concrete but higher than concrete made with either super plasticizer or fly ash. Concrete made with recycled water and either a super plasticizer or fly ash obtained compressive strengths higher than the control. Plain concrete made with recycled water only showed a negative effect on acid resistance but increased the durability as measured through permeability and sulfate resistance. Without admixtures or additives, an increase in the total solids content of the cement paste resulted in lower compressive strengths and set times shortened by 20 minutes.

Improvements in concrete durability were attributed to fine filler effects and the consequent reduced capillary water absorption and porosity found in the denser cement matrix. The accelerated hydration reaction was attributed to the high alkalinity of the recycled water.

2.4 PARTICLE PACKING EFFECTS

A possible reason as to the increased strength properties observed in cement and concrete made with recycled water could be particle packing effects. For a case where all cement particles are of uniform size and shape, the ideal packing configuration of its crystalline structure is a close-packed structure. Even for this ideal packing configuration, however, gaps are still present between the particles. In this simple and homogeneous example, introducing a secondary particle size small enough to fill the voids created would create an overall denser matrix and higher packing factor of the overall structure (Allen 1999). In reality, cement particles contain a distribution of particle sizes, which further complicates the packing scheme. A supplementary cementitious material with a wide and varying particle size distribution can exhibit similar particle packing effects on concrete strength properties by increasing the density of the mortar matrix. Likewise, supplementary cementitious materials substantially finer than cement particles are capable of increasing mortar matrix density by filling gaps created in the mortar matrix.

Supplementary cementitious materials have been known to reduce the overall porosity of mortar, sometimes upwards of 35% (Brooks et al 2011) as well as reduce both the mean and average pore size of the mortars to upwards of 80%. Metakaolin, however, proved to be a more effective pore filler than both fly ash and blast furnace slag. Supplementary cementitious materials with a smaller median and average pore diameter lead to an overall reduction in mortar pore size. The inclusion of supplementary cementitious material was also found to significantly reduce macropores (>50 nm) and increase the number of mesopores (<50 nm). Smaller sized supplementary cementitious particles more easily fill larger macropores and decrease the pore size distribution. Likewise, supplementary cementitious materials can contribute filler effects, which increase concrete strength. As described before, filler effects occur when the

supplementary material has a smaller average particle size than the cement particles and more easily fills voids within the paste to create an overall denser matrix. The increase in compressive strength contributed by filler effects was found to increase as the particle size of the supplementary materials decreased, thus increasing the overall particle size distribution.

Particle packing effects have been reported with other concrete additives, such as rice husk ash. In a study by Bui et al, the effects of rice husk ash on concrete properties were investigated. It was concluded that the relative strength of the concrete increased when rice husk ash was used, if the cement particle size was coarser. This led to the conclusion that the larger size discrepancy between the cement particles and the rice husk ash particles increased the strength by decreasing porosity and improving the particle packing of the structure (Siddique 2008).

2.5 EFFECTS OF HYDRATION

Hydration is a key mechanism in the strength gain, hardening and setting of Portland cement. Hydration is, by definition, the combination of water with an anhydrous material to produce a hydrate. This process is complicated in cement hydration by the fact that there are several compounds and hydration processes occur both in series and parallel. Initial hydration occurs when the two calcium silicate compounds in Portland cement (primarily C_3S) are hydrated by water and form calcium hydroxide and calcium silicate hydrate (C-S-H). It is this calcium silicate hydrate that hardens the mortar matrix by bonding to other unhydrated cement particles, fine aggregate, and coarse aggregates (Kosmatka 2002). The rate of Portland cement hydration most directly depends on the rate of dissolution of the materials, the rate of nucleation and

crystal growth of the hydrates to be formed, and the rate of the diffusion of water and ions through the hydrated material already formed.

The short-term strength of the concrete is more directly dependent on the fineness of the cement and increases as the amount of fine particles increases. Long-term strength is more highly dependent on cement composition. Specific surface area is used to quantify the fineness of the cement. The cement hydration and kinematics are affected by the phase composition of cement and foreign particles within crystalline lattices, the particle size distribution of the cement (and overall fineness), the water-cement ratio, the curing temperature, the presence of chemical admixtures, and additives such as fly ash or slag (Lea, 1998).

Expedited hydration could provide an explanation to the early strength gain observed in concrete made with recycled water. This expedited hydration can occur as a result of the composition of the hydrated cement particles present in the recycled water because the presence of hydrated cement particles can accelerate hydration effects. The primary reaction of cement hydration occurs from the conversion of C_3S into C-S-H. Thomas et al (2009) investigated the hypothesis that the inclusion of C-S-H particles into concrete can expedite the hydration reaction by providing nucleation sites for subsequent C_3S reactions. They theorized there is three primary effects of including fully hydrated cement particles. First, the initial nucleation period can be completely reduced because the C-S-H particles provide nucleation sites for further reactions beginning immediately. Secondly, the acceleration of the entire hydration reaction can increase. Third, the total hydration during early nucleation and growth will increase because of the increased nucleation sites.

While the results of the study confirmed these three effects, testing also revealed that the location of hydration sites changed depending on whether or not C-S-H was initially included.

When cement is left to hydrate without the seeding of C-S-H particles, nucleation was found to initiate near particle surfaces whereas the inclusion of C-S-H shifted this hydration location to between the C_3S particles as well in the pore space. By expanding the possible locations of nucleation, this inclusion of C-S-H particles heavily increased the initial rate of hydration. Increasing the number of nucleation sites also resulted in a more homogeneous final microstructure of the hardened concrete with less capillary porosity. Therefore, the inclusion of recycled water (and consequently C-S-H) would be expected to both increase the rate of hydration as well as the overall mortar matrix density.

2.6 CONCLUSIONS

Despite the limited scope of work and highly variable experiments conducted, several trends can be identified from the present work completed thus far. Generally, material parameters measured to give an indication of performance or to categorize the waste water included pH measurements, total solids contents, and chloride and sulfate contents. Most of the time the measured performance was compared against either ASTM C1602 or EN 1008 requirements, thus the solids, chloride, and sulfate contents fulfilled the criteria outlined. Wash water was found to be highly alkaline with pH measurements exceeding 11.0.

In the experiments conducted, wash water was used as a replacement for mixing water. The use of wash water usually decreased set time and increased the rate of hydration. Increasing the amount of wash water used exacerbated this effect while decreasing workability. If the w/cm was held constant, it was found that workability severely decreased with increasing concentrations of

wash water. If more water was added to improve workability, strength decreased as expected from the increasing w/cm.

Introducing stabilizers to control the workability as a result of the increased wash water further increased the variability in the performance. Admixtures can be used to control the set time and workability but the strength results are inconsistent. Without the use of admixtures, early strengths usually increased. This can be attributed to either the increased rate of hydration or particle packing affects. However, 28-day strengths were much more inconsistent without significant trends present. Most of the concrete produced using wash water, excluding those with extreme replacement levels, fell within the strength requirements outlined in ASTM C1602 (compressive strength must be at least 90% of the control mixture compressive strength).

There is clearly a need for quantifying waste water material parameters and correlating these measurements with concrete performance, given the wide variability of water sources and composition. Results thus far indicate that a correlation should exist between these parameters if it is possible to reduce the scatter historically found in the data. Likewise, there is a need for additional work to account for the fines in the wash water as additional cementitious material. This can ultimately indicate that a solids content exceeding the limits outlined in the ASTM C1602 specification can still produce consistent, acceptable results.

3.0 EXPERIMENTAL INVESTIGATION

The experimental investigation of the recycled fines was divided into two primary sections: an initial materials characterization of the recycled fines and the mortar mixture properties. This included 3- and 28-day compressive strength testing and set time testing. The results of this experimental investigation were ultimately used to build prediction models for the performance of concrete. First, the sources of the recycled fines will be discussed followed by the testing procedures.

3.1 SELECTION OF RECYCLED FINES SOURCES

The behavior and quality of recycled concrete fines as a cementitious replacement material varies widely based on many variables relating to material source. A preliminary division for characterizing fines is based directly on the source of the waste water: wash out water from ready-mix concrete trucks (wash-out fines, or WOF) or grinding fines from pavement maintenance operations (grinding fines, or GF). Wash out fines were produced from water used to wash out ready-mix concrete trucks and were collected from settling ponds or recirculation systems found at ready-mix concrete plants. Grinding fines were produced from a variety of pavement maintenance and construction activities that include saw cutting joints in freshly

placed pavement, and diamond grinding and grooving, which can occur in freshly hardened pavement or as a maintenance procedure for aged pavements.

Because the reaction accelerating potential of this recycled water is hypothesized to be related to the unhydrated cement particles, the age of the fines is an important factor for performance predictions. In total, six recycled fines sources were identified for initial testing and model development: three grinding fines and three wash out fines sources. Details of each recycled fines type are given in Table 1. Washout fines were taken from ready mix concrete plants both near Pittsburgh, PA and Seattle, WA. The concrete plant source in Pittsburgh utilizes a three settling pond system wherein all wash-out water is emptied into the first settling pond. After a set amount of time when the largest fines have settled, the water was moved to the second settling pond and the process repeated for this and the third settling pond as well. This project utilized fines from the third settling pond; therefore, of the wash out water available, the sample used should contain the highest amount of small particles. The Seattle wash out fines were obtained from recycled water recirculation systems so the particle size distribution of this material was expected to be greater than that from the Pittsburgh area wash out fines.

Grinding fines sources were identified based on location, including sources from both the Pittsburgh, PA and Seattle, WA areas, and the age of pavement when the sample was taken. The age of the pavement when the diamond grinding was performed could possibly have an effect on the reactivity of the fines. An older pavement would be expected to be more highly carbonated, which could decrease the reactivity of the fines. This could potentially affect whether the fines will expedite the early strength gain through reactivity, or if any strength effects may be a result of filler effects and improved particle packing, as might be seen with a less reactive particle. In

addition, the grinding fines would be expected to contain a higher percentage of rock dust from the grinding operation.

Table 1. Summary of recycled fines used for initial experimental testing.

	Stoneway	Fairchild	I-79	Stoneway Hauser	Bryan 3	Miles-Auburn
Type	Grinding	Grinding	Grinding	Wash out	Wash out	Wash out
Fines number	GF 1	GF 2	GF 3	WOF 1	WOF 2	WOF 3
Source	I-405	Fairchild Air Force Base	I-79	Stoneway Concrete ready mix plant	Bryan Concrete ready mix plant, settling pond 3	Miles Concrete ready mix plant
Location	Seattle, WA	Seattle, WA	Pittsburgh, PA	Renton, WA	Pittsburgh, PA	Near Seattle, WA
Pavement age	> 10 years	new	~10 years	N/A	N/A	N/A
Date collected		Fall 2011	Fall 2011	2009	Summer 2012	Fall 2012
Additional notes		Grinding occurred within days of construction				

3.2 PREPARATION OF RECYCLED FINES

To prepare for testing, each recycled fines source was collected as waste water and then dried at 40°C. This drying was necessary to control the amount of recycled fines used in later material characterizations and mortar testing as a percentage of the mass of cementitious materials. The implementation plan and guidelines, however, will be developed for waste water with unknown concentrations. The samples were considered sufficiently dry when the change in mass did not vary by more than 1% daily. Once dried, the samples were mechanically sieved in a No. 40 sieve

for 8 minutes. This was to ensure that there were no large agglomerates of fines, which would affect mortar consistency, as well as to remove any pebbles or similar debris.

Following the drying and sieving of a complete source of recycled fines, the entire dried and sieved sample was then mixed and divided to ensure uniformity. This mixing was done through quartering and followed the procedure outlined in ASTM C702: Standard Practice for Reducing Samples of Aggregate to Testing Size.

Once a sample had been dried, sieved, and mixed, it was considered ready to be tested by the procedure discussed in the following sections.

3.3 MATERIALS CHARACTERIZATION

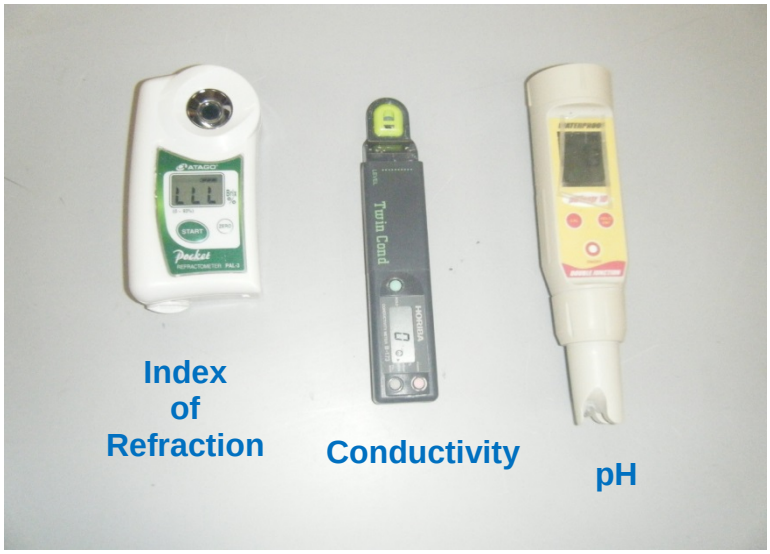
A material characterization testing procedure was developed to build characterization curves based on easily measured parameters in order to quickly describe the waste water. This ensured the applicability of the testing procedure to a water recycling recirculation system with in-line sensors that could be adopted by ready-mix concrete plants. These measured parameters were used to define the fines sources with parameters to be included in the final predictive models. Three material parameters were initially identified to fulfill the criteria of quickly describing possible sources of reactivity of the recycled fines. The index of refraction (IR) was measured to indicate the approximate level of both the suspended solids and dissolved ions. A Brix reading was taken and then used to calculate an IR value since it allowed the measurement to be easily made with a handheld instrument. Conductivity was also measured because of its sensitivity to only dissolved ions and not suspended solids. The combination of the IR and conductivity measurements could then be used to discern between dissolved ions and suspended solids. Then

pH was measured because of its sensitivity to hydroxyl ions and therefore could provide an approximate indication of the rate of reaction of the fines. Ultimately, the three of these parameters combined should provide an indication of the total reactivity of the fines. All three of these parameters can both be measured quickly and using in-line sensors adaptable for plant use, as will be required in further experimental testing to be described later.

Finally, to investigate the possibility of particle size effects, all specimens were scanned in a Microtrac particle size diffraction laser. This equipment provided average size and distribution values for each recycled fines sample.

3.3.1 Materials characterization testing equipment

The equipment used for the material characterization testing is shown in Figure 2 (a) and includes the three handheld sensors for measurement of Brix (to be correlated to Index of Refraction), conductivity, and pH. A milkshake-style mixing stand, typically used in soil testing laboratories, was also used and is shown in Figure 2 (b).



(a) Handheld materials characterization sensors.

(b) Stand mixer.

Figure 2. Equipment used for materials characterization.

The specifications for the handheld equipment used for material characterization are described in Table 2. The index of refraction and conductivity measurements required approximately 2-3 drops of solution each while the pH meter was a probe-type meter.

Table 2. Handheld meters for fines characterization.

Property measured	Sampling	Precision
Brix	2-3 drops in sensor well	0.01%
Conductivity	2-3 drops in sensor well	2% full scale 4 µS/cm up to 199 µS/cm 40 µS/cm, 200-1,999 µS/cm
pH	Immersion in solution	0.1 pH

It is important to note that the measurements taken from the Brix meter were Brix measurements and required conversion into IR for full material characterization. The relationship given as Equation 1 was used for this purpose.

$$IR = 1.33302 + 0.001427193Brix + 0.000005791157Brix^2 \quad (1)$$

A Microtrac diffraction laser was used to gather particle-size effect information for each fines type. The data given by the equipment included a particle size distribution for the scanned particles with average diameters given for each particle size increment. The particle size distribution used was based on the number of particles rather than the mass or volume of the particles, also given as output by the scanning equipment. Given the large amount of data produced for each fines type, it became apparent that a single parameter that could be used to describe the relation of the average particle size as well as the range of particle sizes would be beneficial. Therefore, the span parameter was employed to quantify the relationship between the entire particle range and the median particle size. The span parameter is defined by Equation 2 below. Both the span parameter as well as the d_{50} parameter were found to be significant predictors and were used in model development.

$$\text{Span} = \frac{d_{90} - d_{10}}{d_{50}} \quad (2)$$

Where,

d_{90} = the diameter based on the 90th percentile of the number of the tested particles

d_{50} = the diameter based on the 50th percentile of the number of the tested particles

d_{10} = the diameter based on the 10th percentile of the number of the tested particles

3.3.2 Materials characterization testing procedure

To populate the materials characterization database, six different recycled fines sources were tested. The dried recycled fines were characterized by first placing 518.2 grams of room temperature, de-ionized water with 12.25 grams of recycled fines, representing 1% of the total mass of cementitious material based on the mortar cube mix design to be described in Section 2.4. This mixture was then mixed in a milkshake-style mixing stand shown in Figure 2 (b).

This mixture was then mixed on low speed for four minutes, which was found to be the minimum time required for full mixing and to achieve stabilized material parameter measurements in a preliminary study (Janssen et al 2010). After four minutes of mixing, measurements of the index of refraction, conductivity, and PH were taken using the hand-held meters.

Once measurements were taken, 12.25 grams of fines were added to the mixture and the process was repeated incrementally until a total of 122.5 grams of fines were used, representing 10% of the total mass of cementitious materials based on the quantities for each batch of mortar. The quantities of fines used for each cycle are given in Table 3. This process was repeated at least three times for each recycled fines type until repeatable results were achieved.

Table 3. Recycled fines quantities required for materials characterization.

Test increment	Mass of cementitious materials, percent	Total de-ionized water, grams	Total fines, grams
1	1	518.2	12.25
2	2	518.2	24.50
3	3	518.2	36.75
4	4	518.2	49.00
5	5	518.2	61.25
6	6	518.2	73.50
7	7	518.2	85.75
8	8	518.2	98.00
9	9	518.2	110.25
10	10	518.2	122.50

A small sample (approximately 20 g) of each recycled fines source was mixed with deionized water and placed in the Microtrac diffraction laser for particle size characterization. The particle size analysis obtained for each sample was recorded. This process was repeated until three consistent trials between any single fines source was obtained.

3.4 MORTAR TESTING

Mortar mixtures were then prepared to determine both early-age and long-term compressive strengths as well as initial set times. A series of mortar mixtures were proportioned and prepared with a constant fines content (by mass), which included Portland cement and dried recycled

fines, and in some cases, a cementitious replacement material. A total of seven mortar mixtures were tested for each set of recycled fines. Both slag and Class F fly ash were used as cementitious replacement materials in the mixtures at three different replacement levels for each. The replacement levels were chosen in order to provide a maximum range for standard usage of the cementitious replacement materials in standard practice. The mill sheets for both materials are included in Appendix A. Slag mixtures consisted of 25%, 37.5%, and 50% slag with respect to the total mass of cementitious material and fly ash mixtures consisted of 10%, 20%, and 30% fly ash. A control mixture, containing no recycled fines, was also made for each replacement type.

Four different mixtures were then cast for each of the seven mortar mixture designs with different percentages of recycled fines as a percentage of mass of total cementitious replacement material: 0%, 2.5%, 5%, and 7.5%. It is important to note that the recycled concrete fines were measured as a replacement of the cementitious materials by mass and were included in the mixtures as a dried powder as opposed to inclusion as waste water. This ensured a consistent water to powder (defined as cementitious materials plus the recycled powder) ratio of 0.42 for all mixtures. It is also important to note that there is a significant effect from the additional potential cementitious material in the solids. In order to illustrate this relationship, the Duff-Abrams relationship between strength and water cementitious material ratio will be used and is given in Equation 3 (Mindess et al 2002).

$$\text{Compressive strength, psi} = \frac{A}{B^{1.5(w/cm)}} \tag{3}$$

Where,

A = 14,000 psi, a typical empirical constant.

B = 4, based on typical cement properties.

The w/cm was held constant at 0.4 for the comparison and the percent solids were varied between 0 and 20%, assuming that all of the percent solids were cementitious materials. The plot of strength as a function of percent solids of the recycled water is given in Figure 3 below. Therefore, this can allow for visualizing the effect on compressive strength if all of the recycled fines were composed of un-hydrated cement particles and the mixture proportions are based on w/cm ratio rather than the ratio of water to powder.

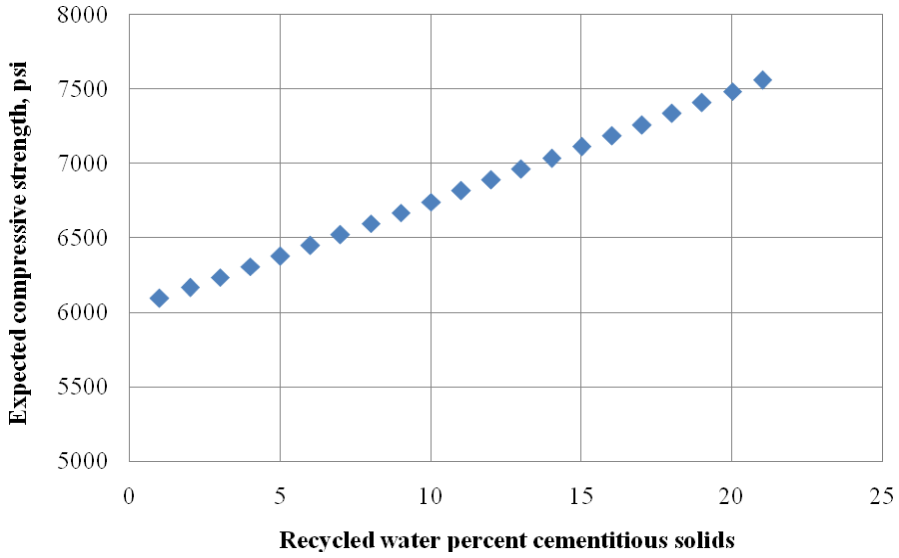


Figure 3. Expected effect on compressive strength as a function of percent solids in recycled water.

It can be seen that a steady linear relationship exists between the expected compressive strength and the percentage solids in recycled waste water. An outline of the mixture designs by mass is given in Table 4.

Table 4. Mortar mixture designs, mass for each component in grams.

		Percent fines			
		0	2.5	5	7.5
Control	Cement	1,225.0	1,194.4	1,163.8	1,133.1
	Sand	1,947.0	1,947.0	1,947.0	1,947.0
	Water	518.2	518.2	518.2	518.2
	Fines	0.0	30.6	61.3	91.9
50% Slag	Cement	612.5	597.2	581.9	566.6
	Slag	612.5	597.2	581.9	566.6
	Sand	1,947.0	1,947.0	1,947.0	1,947.0
	Water	518.2	518.2	518.2	518.2
	Fines	0.0	30.6	61.3	91.9
37.5% Slag	Cement	765.6	746.5	727.3	708.2
	Slag	459.4	447.9	436.4	424.9
	Sand	1,947.0	1,947.0	1,947.0	1,947.0
	Water	518.2	518.2	518.2	518.2
	Fines	0.0	30.6	61.3	91.9
25% Slag	Cement	918.8	895.8	872.8	849.8
	Slag	306.3	298.6	290.9	283.3
	Sand	1,947.0	1,947.0	1,947.0	1,947.0
	Water	518.2	518.2	518.2	518.2
	Fines	0.0	30.6	61.3	91.9
30% Fly Ash	Cement	857.5	836.1	814.6	793.2
	Fly Ash	367.5	358.3	349.1	339.9
	Sand	1,947.0	1,947.0	1,947.0	1,947.0
	Water	518.2	518.2	518.2	518.2
	Fines	0.0	30.6	61.3	91.9
20% Fly Ash	Cement	980.0	955.5	931.0	906.5
	Fly Ash	245	238.9	232.8	226.6
	Sand	1,947.0	1,947.0	1,947.0	1,947.0
	Water	518.2	518.2	518.2	518.2
	Fines	0.0	30.6	61.3	91.9
10% Fly Ash	Cement	1,102.5	1,074.9	1,047.4	1,019.8
	Fly Ash	112.5	119.4	116.4	113.3
	Sand	1,947.0	1,947.0	1,947.0	1,947.0
	Water	518.2	518.2	518.2	518.2
	Fines	0.0	30.6	61.3	91.9

On each mixing day, four mixtures with a single type of waste fines at the four replacement levels were cast using a single percentage of the cementitious replacement type. Six 2 in x 2 in mortar cubes were made from each batch according to the specification ASTM C109: Standard Test Method for Compressive Strength of Hydraulic Cement Mortars. The short-term (3-days) compressive strengths were measured for three specimens and the long-term (28-days) compressive strengths were measured for the remaining three. The compressive strengths were measured, as shown in Figure 4 below, and load was continuously applied until failure, as specified in ASTM C109.

Figure 4. Compression machine used for mortar cube samples.

Mortar set time was measured with a Vicat testing apparatus, as shown in Figure 5 and in accordance with ASTM C807-08: Standard Test Method for Time of Setting of Hydraulic Cement Mortar by Modified Vicat Needle. This included casting a cylindrical mortar sample in

two lifts and using the penetration needle to measure depth of penetration every 30 minutes and increasing readings to every 10 minutes when penetrations were less than 40 mm. The mortar sample is considered to have reached initial set once the penetration measurements are less than 10 mm.

Figure 5. Vicat apparatus for mortar set time testing.

4.0 RESULTS

4.1 MATERIALS CHARACTERIZATION RESULTS

The materials characterization procedure was repeated for each fines type until consistent results from three trials were obtained. Results from these three trials were then averaged to obtain a representative value for each of the three material parameter measurements. The complete set of plots for results for all six sets of fines is given in Appendix B while the complete set of numerical results is given in Appendix C. Only the results and calculations for sample number 2 of the wash out fines (WOF 2) are presented here for the sake of brevity but the calculations for the other five fines types were completed in the same manner. Results from the three replicated trials for the three material characterization parameter measurements are given in Figure 6 for all six recycled fines samples.

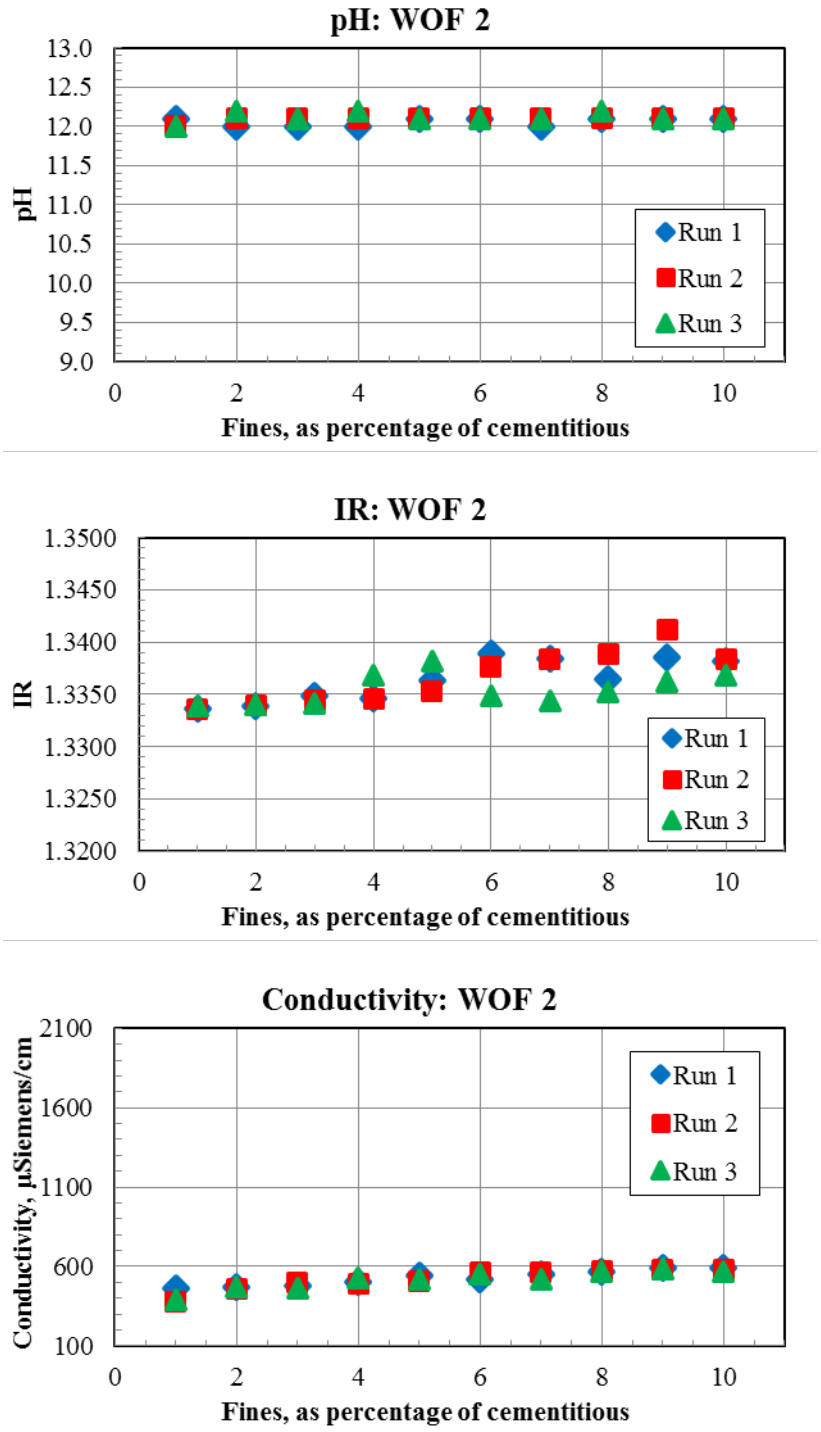


Figure 6. Materials characterization parameter plots for WOF 2.

Several trends can be observed from the plots of these material parameters for WOF 2. The pH value is relatively constant and does not vary with concentration. The conductivity

increases slightly with increasing fines content and the index of refraction value appears to be most directly related to the concentration of recycled fines. Overall, the values appear to be repeatable and consistent; therefore, the values obtained from these three trials are then averaged to create a single representative trial for the specific set of fines. The average pH, conductivity, and IR (calculated from measured Brix values) for WOF 2 are given in Table 5.

Table 5. Average material characterization measurements for WOF 2.

Test	Fines, grams	Average from three trials		
		pH	IR	Conductivity, μS/cm
1	12.25	12.0	1.33369	410.00
2	24.50	12.1	1.33393	466.67
3	36.75	12.1	1.33450	480.00
4	49.00	12.1	1.33532	506.67
5	61.25	12.1	1.33662	523.33
6	73.50	12.1	1.33716	543.33
7	85.75	12.1	1.33701	543.33
8	98.00	12.1	1.33682	570.00
9	110.25	12.1	1.33863	583.33
10	122.50	12.1	1.33779	580.00

All relationships were assumed to be roughly linear based on observed trends. The slope and intercept from a linear fit for each material parameter is then calculated along with a corresponding R^2 and are given for the WOF 2 parameters in Table 6.

Table 6. Linear fit results for WO2 fines.

	pH	IR	Conductivity, μS/cm
Slope	0.00505	0.000528	17.74
Intercept	12.06	1.333245	423.11
Standard error	0.0241	0.0006	15.71
R^2	0.31	0.89	0.93

It can be seen from the R^2 that the correlation for pH does not fit well. This is attributed to the relatively constant values with low variation, as evidenced by the low standard error. These slope and intercept are then used to define linear plots for each parameter. This procedure is then repeated for each of the six sets of recycled fines to obtain representative linear plots for the six fines samples between the range of 1% and 10% of total cementitious material by mass. These percentages correspond to the inclusion of between 12.25 grams of fines and 122.5 grams of fines. The trends between the different types of fines can now be compared, as shown in the plots given in Figure 7 to Figure 9. All wash out fines are shown with dashed lines and hollow markers while grinding fines are designated with solid lines and markers.

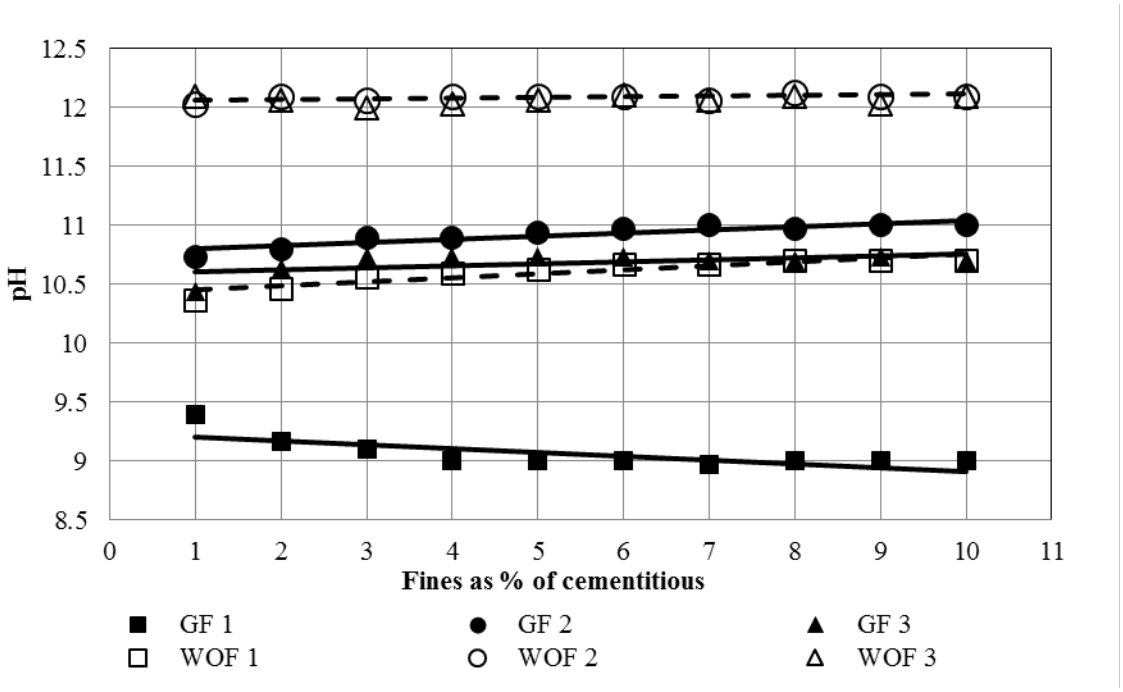


Figure 7. pH readings as a function of fines concentration.

Certain trends can be observed from these plots. The pH, which is expected to provide an indication of reactivity, appears to be quite linear with a relatively small slope. The largest differentiation between the different fines types is the magnitude of the pH rather than the degree of slope. This is evidenced from the data presented in Table 7 where the slopes for the pH lines are all close to zero. Both WOF 2 and WOF 3 have a similar pH reading close to 12.0 and that is relatively constant regardless of concentration. These two fines sources are both young wash out fines (from 2011 and 2012, respectively). Additionally, the pH readings of WOF 1 and both GF 2 and GF 3 are similar. However, the sources of these three fines are less similar. WOF 1 was the oldest wash out fines (from 2009) and therefore possibly affected by carbonation. GF 2 was produced from grinding a new concrete pavement immediately following construction and GF 3 was produced from maintenance diamond grinding a 10-year old pavement. Finally, GF 1 behaved completely differently. The pH decreased with increasing concentration and was the lowest in magnitude of all of the fines sources.

The pH is expected to be related to the ability of the fines to increase the rate of hydration. From this materials characterization, it would be expected that WOF 3 and WOF 2, which are the two youngest wash out fines, would increase the rate of hydration the most, thus leading to the highest decrease in set time and possibly the highest increase in short-term compressive strength.

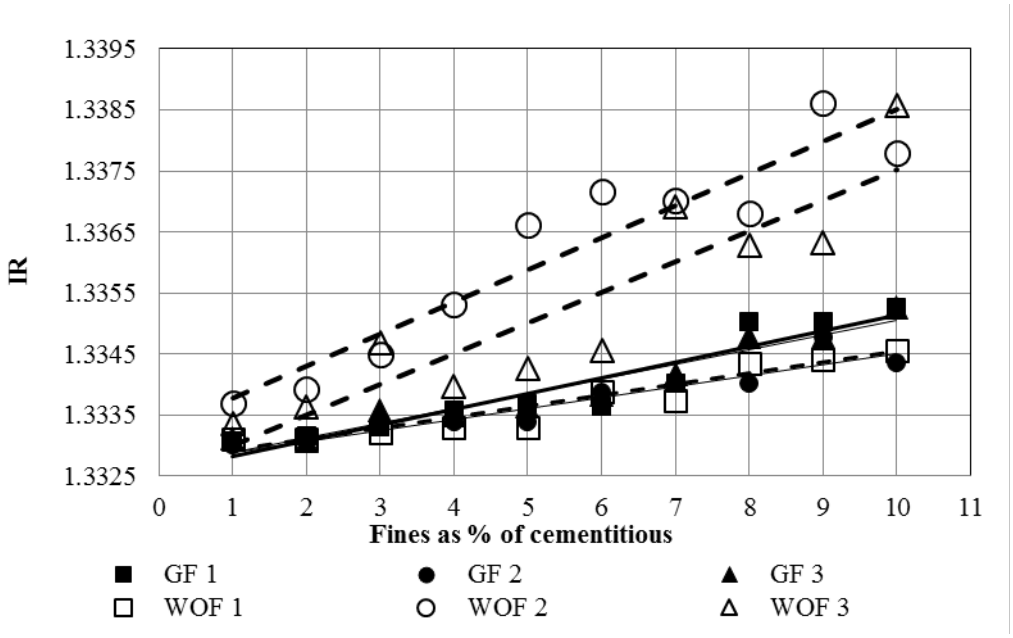


Figure 8. IR measurements as a function of fines concentration.

The index of refraction readings all increased with increasing concentration as expected, because the index of refraction will approximately indicate both the dissolved and suspended solids content. Again, the WOF 2 and the WOF 3 have similar trends but different magnitudes. WOF 2 overall had a higher index of refraction than WOF 3 but both had nearly identical slopes. GF 1 and GF 3 were nearly identical in their behavior as well; however, the index of refraction was significantly lower than WOF 3 and WOF 2. WOF 1 and the GF 2 had the lowest index of refraction and also had nearly identical results.

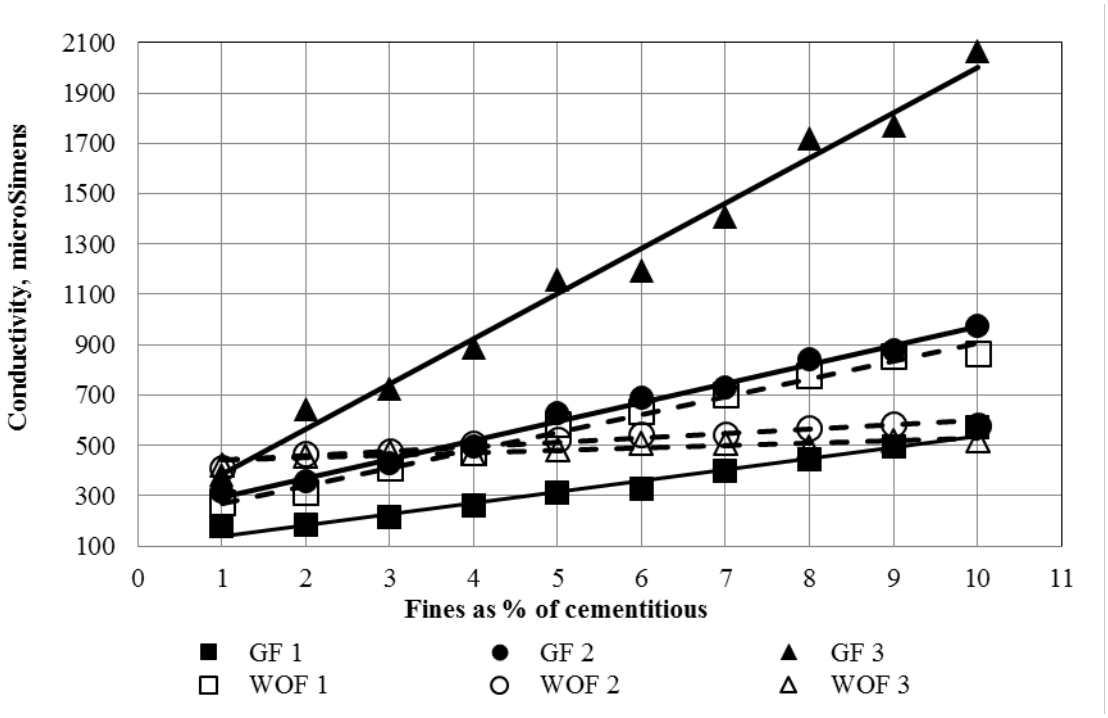


Figure 9. Conductivity readings as a function of fines concentration.

The conductivity readings are expected to give an indication of dissolved ions only. Again, the two youngest wash out fines, the WOF 2 and the WOF 3, behave similarly with a relatively small slope and an overall low conductivity ranging only between 450 and 600 μS/cm. Similar to the pH readings, GF 2 and WOF 1 again behaved similarly for this parameter, with a steeper slope than the other two wash out fines. The approximate range for WOF 1 and GF 2 conductivity readings falls between 300 and 900 μS/cm. GF 3, acquired from the older pavement, has extremely high conductivity readings with a very steep slope: increasing substantially with increasing concentration. The range for the conductivity falls between 400 and 2,000 μS/cm. The primary difference between GF 3 and GF 2 is the age of the pavement, which could explain the difference in dissolved ions. Of the six fines types tested, GF 3 is from a 10 year old pavement. GF 1, however, which was from the oldest pavement but had a similar trend to GF 2 and WOF 1, results but lower in magnitude. The dissolved ions content would be expected to be similar to GF 2.

Since the index of refraction measurements provide an indication of total solids content while the conductivity measurements indicate dissolved solids only (not suspended solids), the two measurements must be considered together. From the measurements, it appears that the two youngest washout fines, WOF 2 and WOF 3, have the highest number of total solids, which increases with concentration. However, the conductivity measurements indicate a moderate amount of dissolved solids, which increased very little with increasing concentration. Therefore, for the young wash out fines, the amount of suspended solids increases with increasing concentration but the dissolved ions does not. GF 2 and WOF 1 exhibited similar and consistent behavior: both the dissolved and total solids increased moderately with increasing fines concentration. GF 1 had a similar increasing trend but was less gradual and would therefore overall contain fewer solids than the other fines types. Finally, GF 3 overall contained a moderate amount of total solids but by far the highest number of dissolved solids. This discrepancy indicates that GF 3 had a relatively low number of suspended solids. From the plots in Figures 7, 8, and 9, relative levels can be established and trends can be observed.

Table 7 gives a summary of the trends and similarities discussed above between the three material parameters. The relative levels of these recycled fines samples are given in Table 7 based on the trends seen in Figures 7, 8, and 9.

Table 7. Summary of trends and similarities between recycled fines samples.

	pH	Conductivity	Index of refraction
Low	GF 1	GF 1	GF 2
			WOF 1
Medium low	GF 2	WOF 2	GF 1
	WOF 1	WOF 3	
Medium high	GF 3	GF 2	GF 3
		WOF 1	
High	WOF 2	GF 3	WOF 2
	WOF 3		WOF 3

Average values for each of the three materials characterization measurements previously discussed were then calculated for each fines type and are provided in Table 8.

Table 8. Materials characterization results for recycled fines.

		Slope	Intercept	Standard Error	R^2
GF 1	pH	-0.033	9.24	0.0945	0.55
	Conductivity	44.06	96.67	22.85	0.97
	IR	0.0003	1.33	0.00028	0.90
GF 2	pH	0.027	10.77	0.0408	0.82
	Conductivity	75.39	220	22.88	0.99
	IR	0.000181	1.33	0.0002	0.89
GF 3	pH	0.0172	10.59	0.082	0.31
	Conductivity	179.4	208.7	65.24	0.99
	IR	0.00024	1.332	0.0002	0.93
WOF 1	pH	0.034	10.42	0.0501	0.82
	Conductivity	70.65	200.4	23.35	0.99
	IR	0.00018	1.33	0.0002	0.91
WOF 2	pH	0.0051	12.06	0.0241	0.31
	Conductivity	17.74	423.1	15.71	0.93
	IR	0.0005	1.33	0.0006	0.89
WOF 3	pH	0.0024	12.05	0.0368	0.043
	Conductivity	9.62	431.8	12.90	0.85
	IR	0.0005	1.33	0.0008	0.81

From this data, it can be seen that linear trends fit the relationships quite well. The low R^2 seen for pH relationships can be attributed to the relatively constant readings, as shown by the relatively low slope (near zero). Otherwise, the linear trends fit quite well, as evidenced by the high R^2 and the relatively low standard error for each case.

The particle size distribution based on the number of particles was then plotted for each of the six fines type investigated with and is shown in Figure 10.

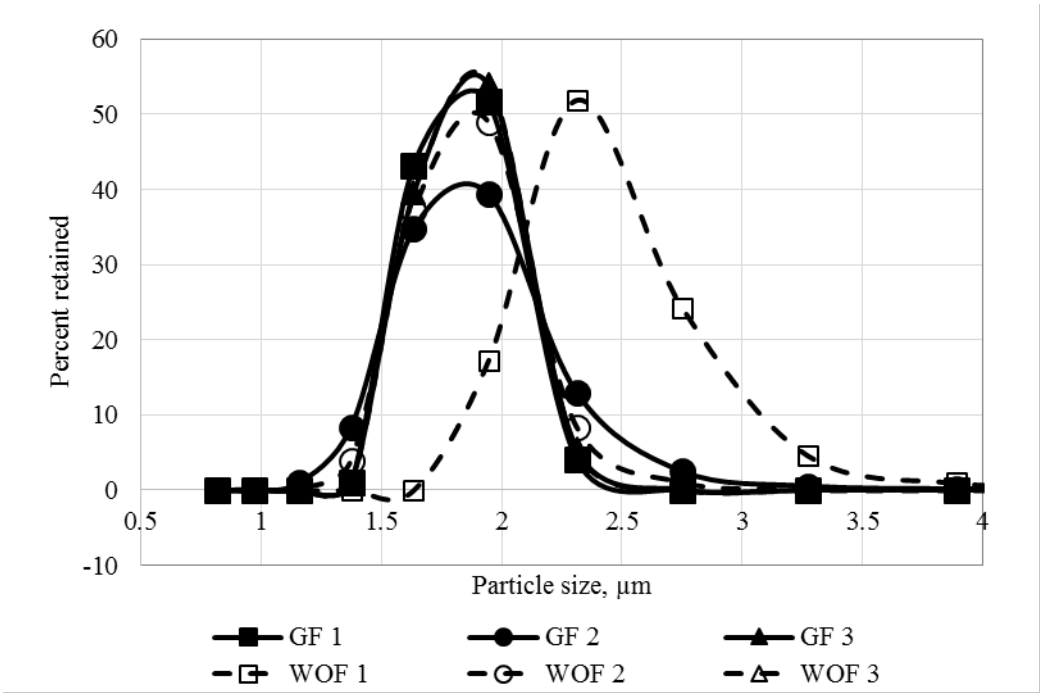


Figure 10. Particle size measurements as a function of fines concentration based on number of particles.

From the plot, it can be seen that the particles have similar, but slightly different, distributions. Most notably, the WOF 3 sample has a slightly larger average particle size. The GF 2 sample has a wider distribution than the other fines types and WOF 2 has a slightly wider distribution than the other fines samples. The particle size distributions of GF 1, WOF 3, and GF 3 are very similar. The span and d_{50} values were then calculated from the particle size distributions from Figure 10 and the equations previously mentioned. All calculated values are presented in Table 9 below.

Table 9. Particle size results for recycled fines based on number of particles.

Fines Type	Span	d_{50}
GF 1	0.270	1.66
GF 2	0.271	1.67
GF 3	0.273	1.68
WOF 1	0.253	2.17
WOF 2	0.245	1.68
WOF 3	0.247	1.68

4.2 MORTAR MIXTURE RESULTS

Mortar mixtures were prepared to test three criteria: 3-day compressive strength, 28-day compressive strength, and set time. Compressive strength data was obtained using mortar cubes, which were tested in accordance with ASTM C109 to obtain both early (3-day) and long-term (28-day) compressive strengths. Both were reported as an absolute strength and as a percentage of the control strength. The control strength used for percentage calculations was the strength obtained for plain Portland cement mortar mixtures without supplementary cementitious replacement material and without recycled fines. Thus, all subsequent mixtures can be reported as a percentage of these values for both 3-day and 28-day compressive strengths.

The intent of testing these mortar properties is to obtain data to construct three predictive models: one for the change in set time from the control mixture, one for 3-day strength as a percentage of the control strength, and one for 28-day strength as a percentage of the control strength. These three parameters were selected because they are the stated criteria in ASTM C1602: Standard Specification for Mixing Water Used in the Production of Hydraulic Cement Concrete. This specification states that any mortar made with a different water source cannot have a strength value less than 90% of the control strength or the water cannot be used. Therefore, these three parameters must be evaluated when considering the use of a different water source. The complete set of data collected during testing is given in Appendix D. During this proposed initial testing, the solids in the recycled water are treated as a supplementary cementitious material; however, this approach was primarily taken to control workability in order to develop a robust predictive model. For implementation guidelines, the recycled fines will be treated as mixing water; therefore, the ASTM C1602 guidelines would be appropriate.

4.2.1 Set time

The mortar set time was tested through a modified Vicat testing apparatus as detailed in ASTM C807: Standard Test Method for Time of Setting of Hydraulic Cement Mortar by Modified Vicat Needle resulting in data for an initial set time in minutes. In ASTM C1602: Standard Specification for Mixing Water Used in the Production of Hydraulic Cement Concrete, acceptable limits for different water sources are measured as a difference from the set time of a control mix. The specification gives an acceptable range of set times as neither 60 minutes shorter nor 90 minutes longer than the control mixture. Throughout this project, the difference in set time is defined as the revised mortar mixture set time subtracted from the control mixture set time. Because in most cases, the revised mix set time is shorter than the control mixture, these values are presented as negative values.

Therefore, to compare all data and ultimately be able to build a model incorporating data from all seven types of mixtures, including the cementitious replacement materials, the final data set used was the difference from each of the three recycled fines replacement percentages (2.5%, 5%, and 7.5%) from the control mix, thus "normalizing" all results by supplementary cementitious material. This allowed for comparison across all data types and the inclusion of all data into an eventual model.

First, the difference in set time will be considered. Therefore, plots can be presented from the three material characterization parameters: pH, conductivity, and IR against the difference in set time and are shown in Figure 11 to Figure 13 below. The CaO content, given as a percentage and calculated based on known values for the cement, fly ash, and slag, is also considered in the predictions and is plotted against the difference in set time in Figure 14. Finally, the diameter of the 50^{th} percentile particle size based on the number of particles is plotted against the difference

in set time in Figure 15 below. As mentioned earlier, both the d_{50} diameter and the span parameter are used to describe particle size effects. The models are constructed with both parameters and the single particle size parameter that produced a better fit and more stable and robust model was ultimately included. Therefore, only a plot of the d_{50} was produced for the set time model.

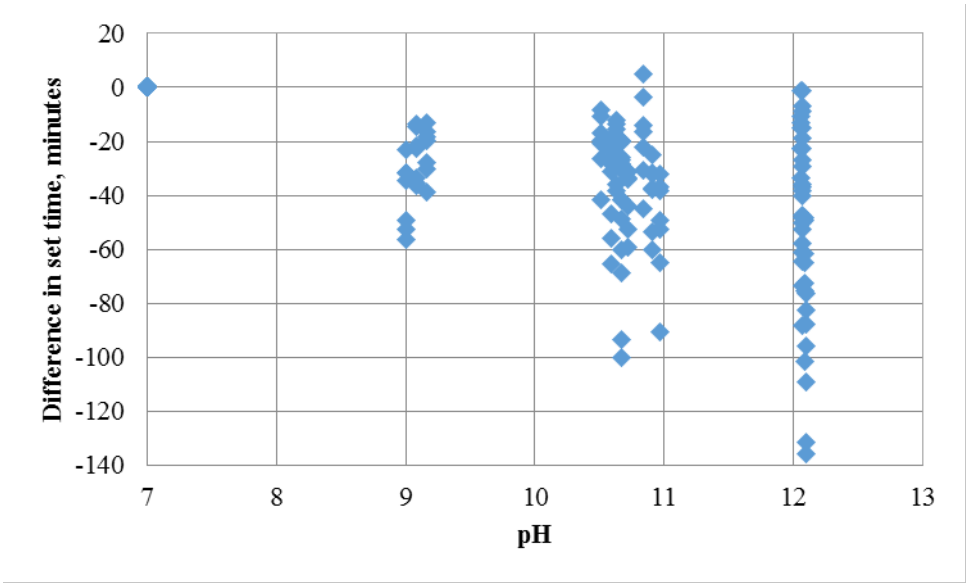


Figure 11. pH versus difference in set time.

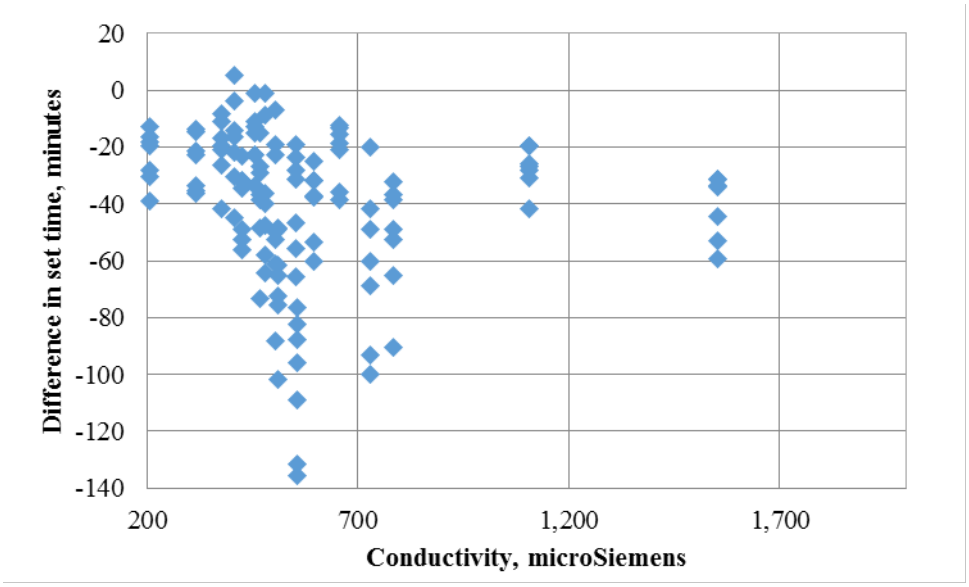


Figure 12. Conductivity versus difference in set time.

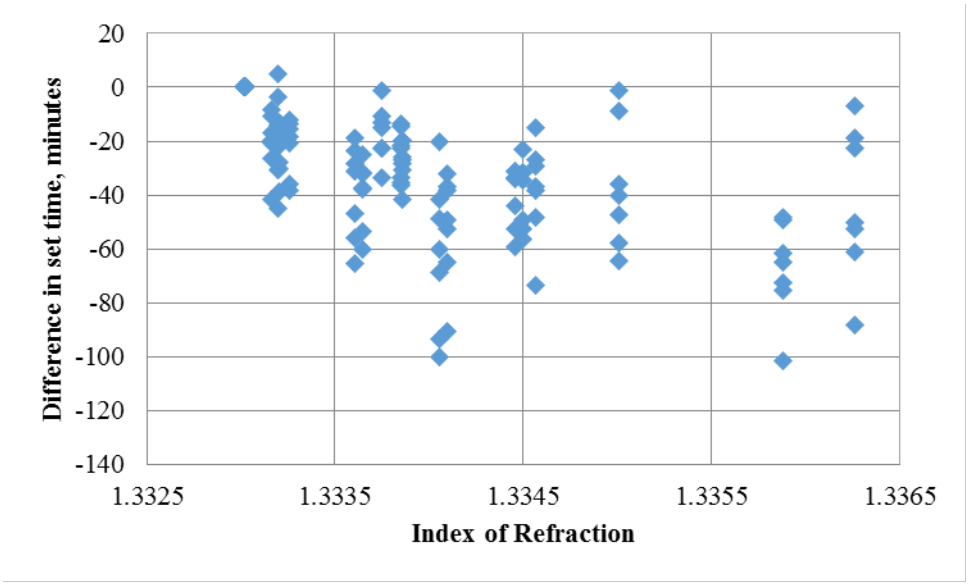


Figure 13. Index of refraction versus difference in set time.

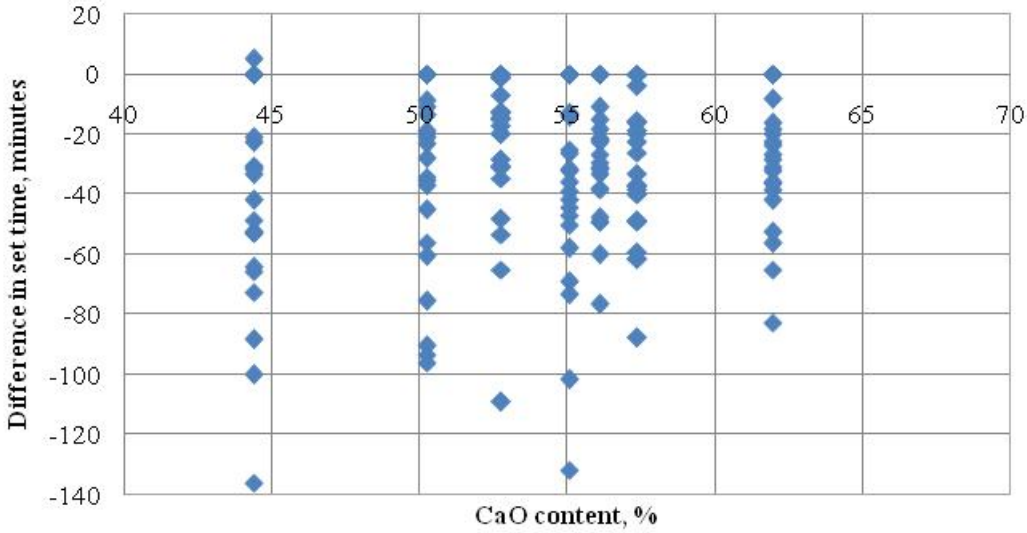


Figure 14. CaO content versus difference in set time**.**

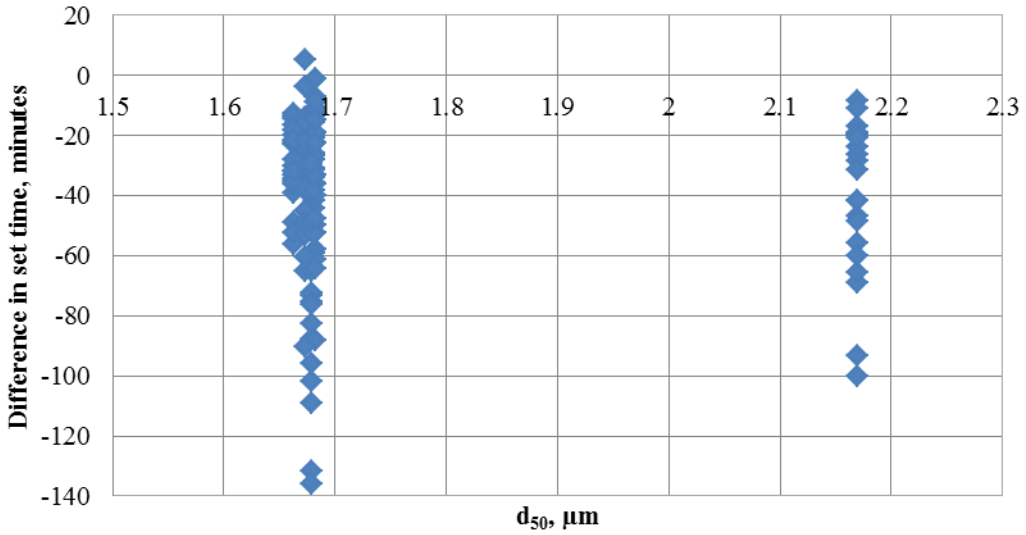


Figure 15. Diameter of 50th percentile particle based on number of particles, d_{50}, versus difference in set time.

While strong trends are not immediately obvious from these relationships, all factors will be considered in building a multiple linear regression, as will be described later.

4.2.2 Compressive strengths

The next phase of the laboratory work consisted of measuring the 3-day and 28-day compressive strengths of mortar cubes. Six cubes were cast for each mixture design (see Table 4), three of which were used for measuring 3-day compressive strengths and the remaining three for 28-day compressive strengths. The mix water used in each of the mortar cube batches was tested for pH, conductivity, and Brix (for IR). The strengths, as a percentage of the control, are shown with respect to these factors in Figures 16 to 18. For additional particle size considerations, the span, as defined in Equation 2, is plotted against the percentage of the control 3-day compressive strength in Figure 19. While compressive strengths are measured for ages of both 3- and 28-days,

the focus of the results remains on early mortar properties. This is consistent with the initial speculation that using the recycled fines as a cementitious replacement material would more greatly affect the 3-day strength and the effect would be greatly lessened for 28-day compressive strengths.

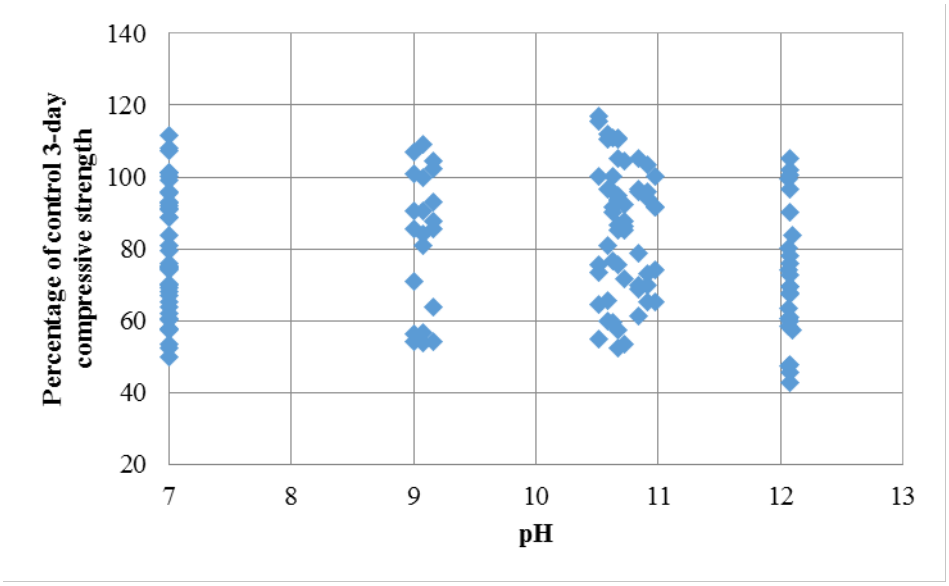


Figure 16. pH versus percentage of control 3-day compressive strength.

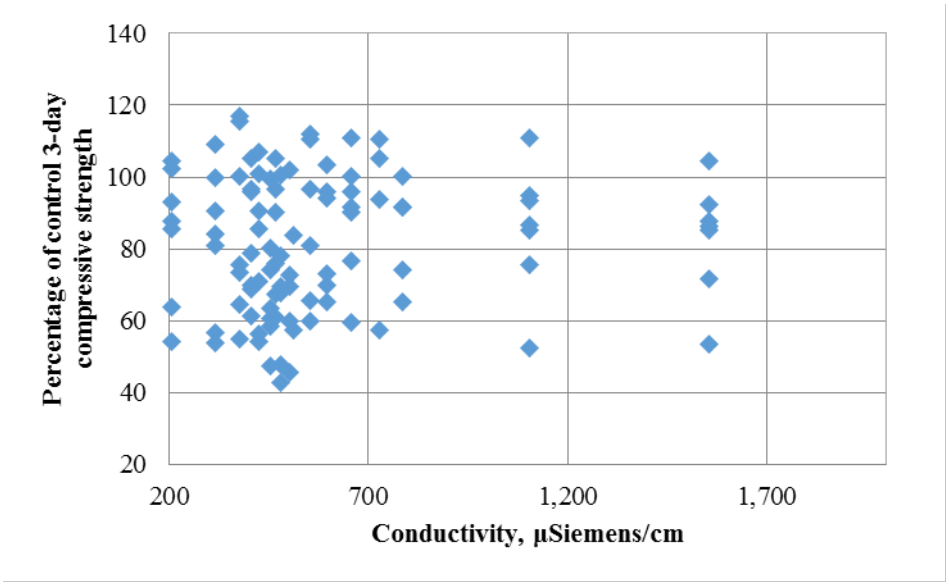


Figure 17. Conductivity versus percentage of control 3-day compressive strength.

Figure 18. Index of refraction versus percentage of control 3-day compressive strength.

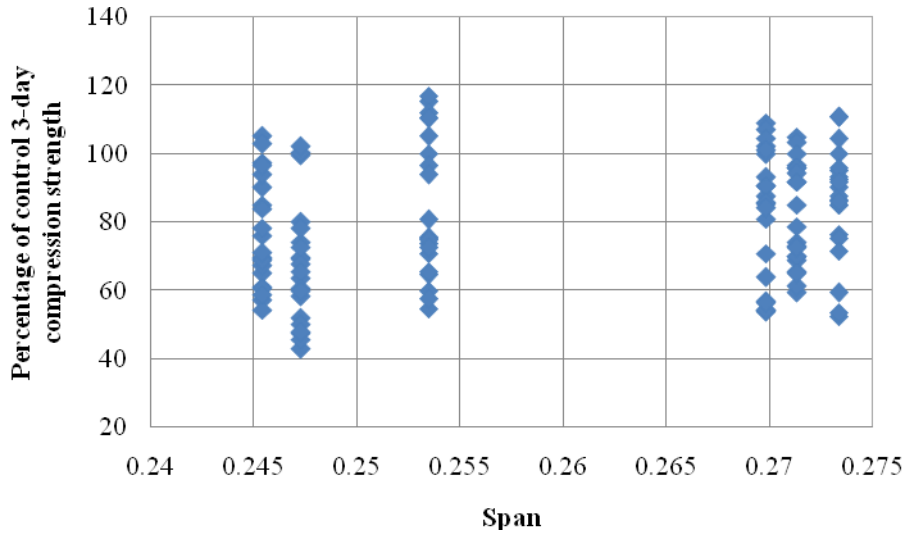


Figure 19. Span versus percentage of control 3-day compressive strength.

The compressive strengths as a percentage of the control are plotted against pH, conductivity, and IR in Figures 20 to 22. Additional particle size effects are also considered and the percentage of control strength is plotted against the span, as defined in Equation 2, in Figure 23.

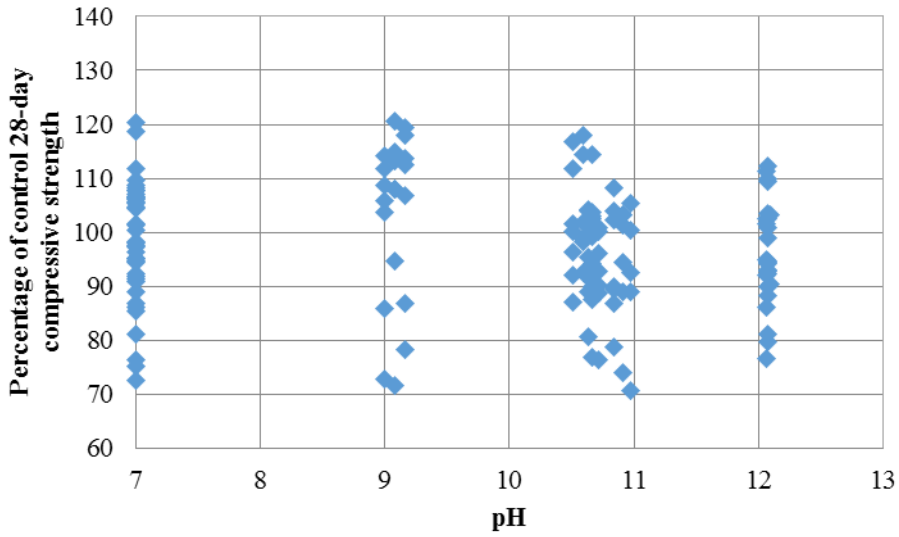


Figure 20. pH versus percentage of control 28-day compressive strength.

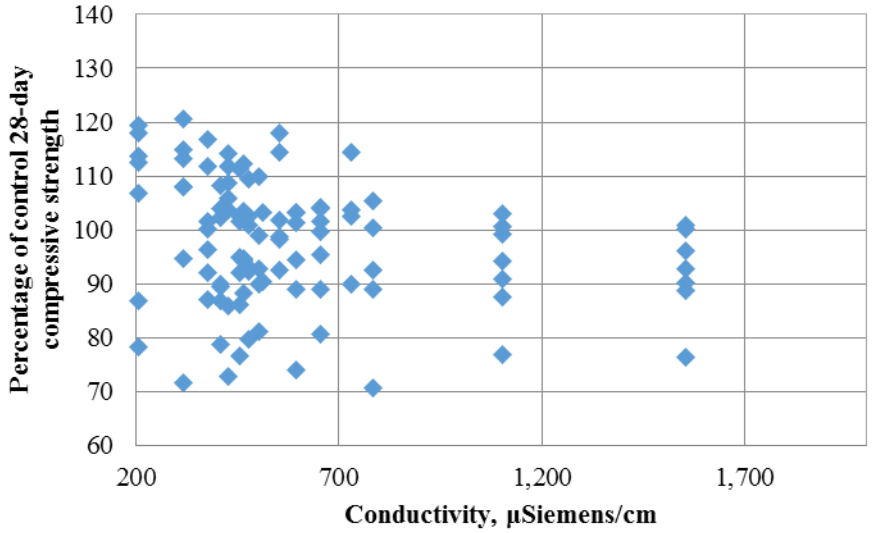


Figure 21. Conductivity versus percentage of control 28-day compressive strength.

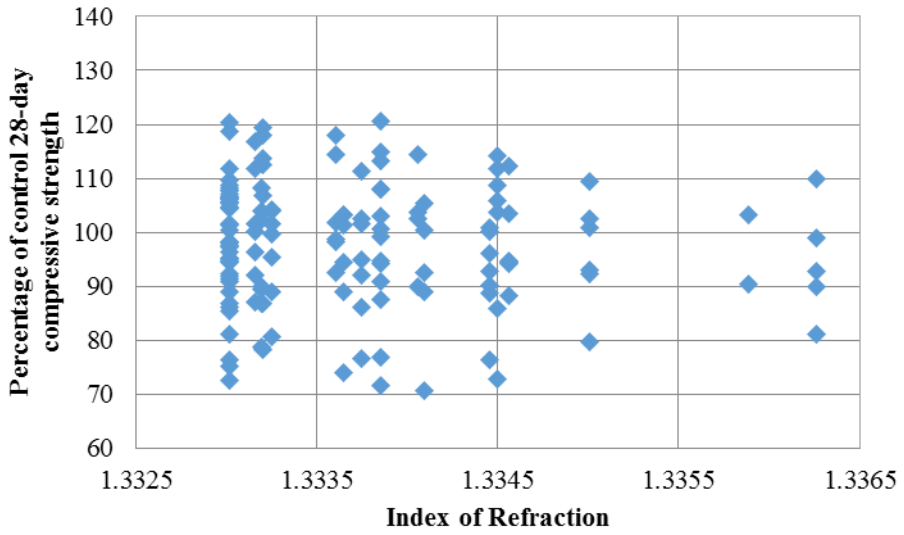


Figure 22. Index of refraction versus percentage of control 28-day compressive strength**.**

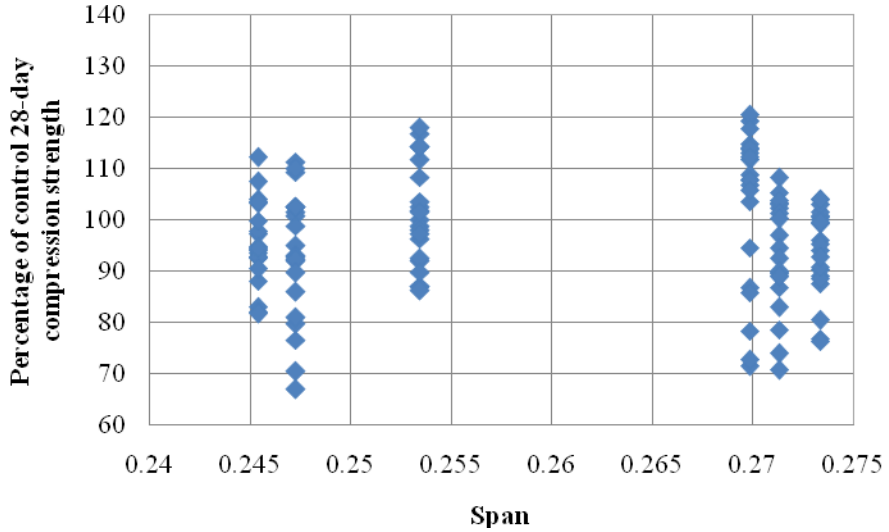


Figure 23. Span versus percentage of control 28-day compressive strength.

Additionally, the mortar samples were normalized to account for the varying levels of different supplementary cementitious materials used in the mix designs. In order to account for this variation, two material-based parameters were considered to normalize the supplementary cementitious material content. First, the CaO content, given as a percentage, was considered.

Secondly, a CaO ratio, defined in Equation 4, was also used to account for differences in the results based on the supplementary cementitious material used.

$$\text{CaO Ratio} = \frac{CaO}{Al_2O_3 + SiO_2} \tag{4}$$

Where,

CaO = percentage content of calcium oxide in the cement or cementitious material

Al_2O_3= percentage content of aluminum oxide in the cement or cementitious material

SiO_2= percentage content of silicon dioxide in the cement or cementitious material

The total number used in the analysis, however, incorporates the varying percentages of cementitious replacement material as well such that the final CaO ratio considered for each mix is using Equation 5.

$$\text{Mix CaORatio} = (F_C)(CaORatio_C) + (F_{SCM})(CaORatio_{SCM}) \tag{5}$$

Where,

F_C= fraction of the total cementitious materials comprised of cement

$CaO\ Ratio_C$= CaO ratio of cement

F_{SCM} = fraction of the total cementitious materials comprised of supplementary cementitious material (either slag or fly ash)

$CaO\ Ratio_{SCM}$ = CaO ratio of the supplementary cementitious material

In order to justify this normalization procedure, the compressive strength must have a linear relationship with the CaO ratio, implying that strength predictions can be made based on the amount and type of supplementary cementitious material. Therefore, the 3-day control strengths can be plotted against the CaO ratio, as shown in Figure 24. It was found that a linear relationship fit the data well with an R^2 of 0.89 and a standard error of 250 psi.

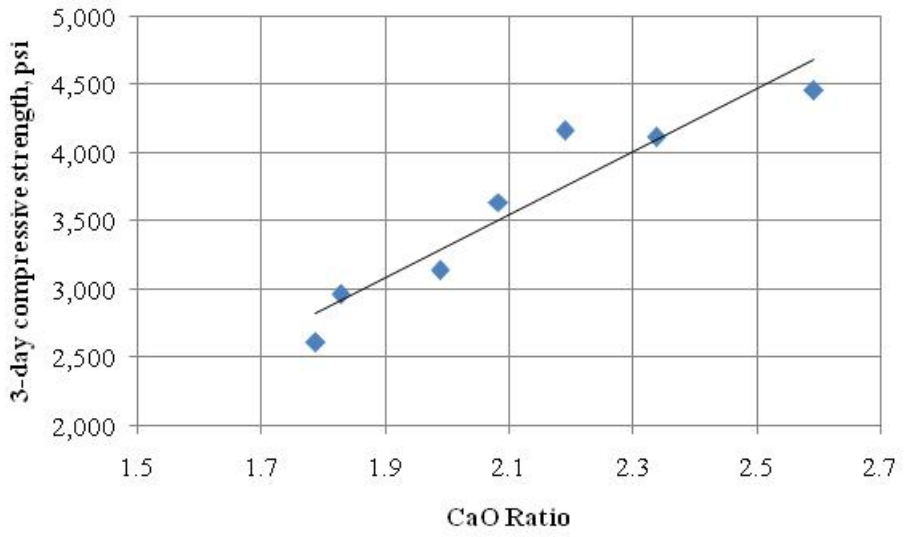


Figure 24. CaO ratio versus control 3-day compressive strength.

The 28-day control strengths are then plotted against the CaO content given as a percentage and shown in Figure 25. This relationship was not as strong as the relationship with early strength and resulted in an R^2 of 0.50 and a standard error of 550 psi. This relationship is expected to be weaker considering that the expected effects from the inclusion of the recycled fines have a greater effect on early strength.

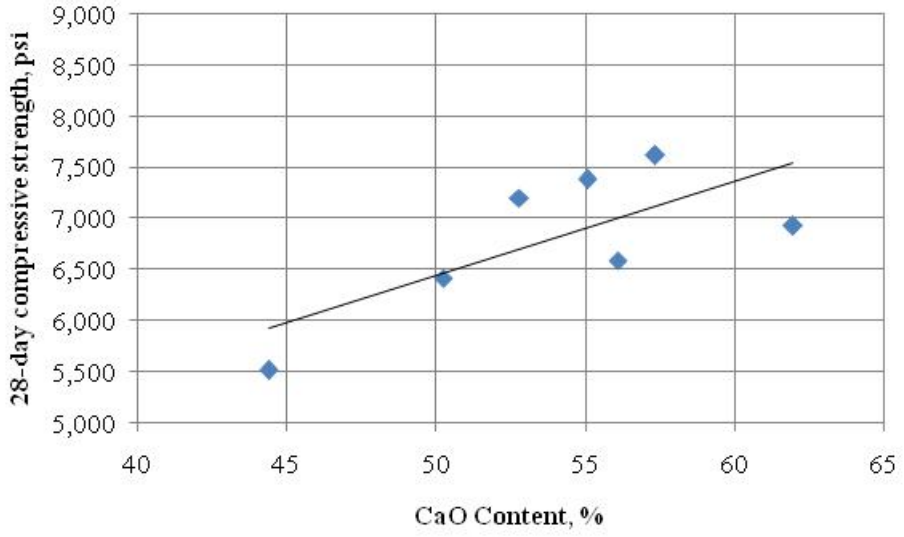


Figure 25. CaO content versus control 28-day compressive strength.

Given that this normalization procedure is valid, both the CaO ratio and the CaO percentage were proportioned, as described by Equation 5. The relationship between the CaO ratio and the percentage of the control 3-day compressive strength is given in Figure 26 and the relationship between the percentage of the 28-day control compressive strength is given in Figure 27.

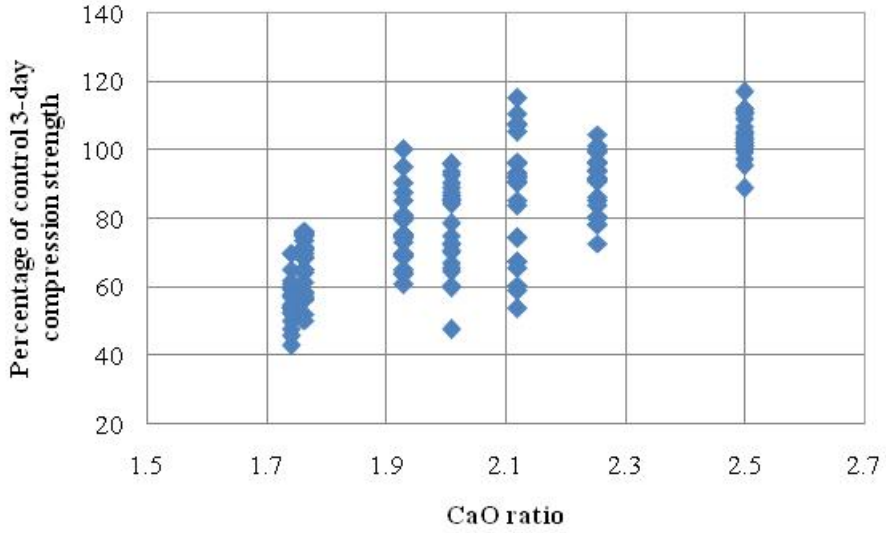


Figure 26. CaO ratio versus percentage of control 3-day compressive strength.

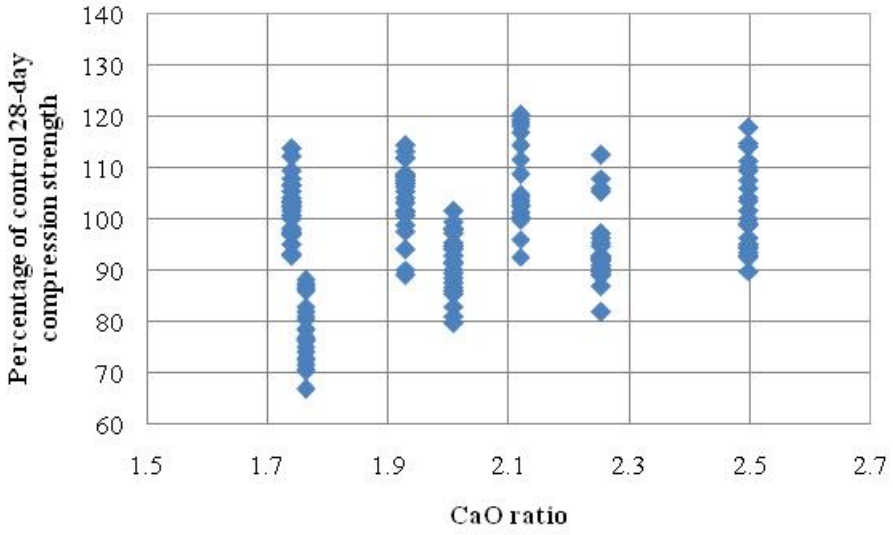


Figure 27. CaO content versus percentage of control 28-day compressive strength.

5.0 DATA ANALYSIS

The laboratory work completed and previously discussed was then used as a database in order to build the intended predictive equations. Because the intent of the project was to predict performance of the mortar using initial measurements of the waste water, the data was organized for regression modeling. Therefore, the predicted performance factor (either the difference in set time or 3- or 28-day strength as a percentage of control strength) would be described as a function of the input parameters selected from the waste water. The process of developing these regression equations will be discussed in this chapter, beginning with sorting the initial data to check for possible outliers and required transformations, and into the development of the final models.

5.1 DATA TRANSFORMATIONS

An initial check for out of range measurements was performed when calculating average compressive strengths from three cubes for each batch, in accordance with the requirements outlined in the ASTM C109 specification. This specification states that out of range specimens should not be considered and of three cubes cast in the same mortar batch, no result should vary by more than 8.7% from the average compressive strength for the three cubes. If a measurement is identified and removed, then neither of the two remaining results should vary by more than

7.6% from the average of the two specimens. This specification requirement provided the first method of identifying unacceptable measurements. All data points were used in the development of all six models. When plotted, no substantial outliers were observed, and the requirement from the mortar cube specification was the only method of identifying unacceptable measurements that was employed.

Initially, the raw compressive strengths were plotted against each for the three material parameter predictors (pH, IR, and conductivity) individually for each of the seven mix designs to look for approximate trends. The lack of linearity of the data indicated that a linear regression model would not fit the data well. Two options emerged: either a multiple nonlinear regression model could be used or the raw data could be transformed using nonlinear functions and the transformed data could then be used in a linear regression model. Data transformation in a linear regression model was deemed simpler and was therefore tried first. Nonlinear regression analysis was attempted, but as only one variable was transformed for each final model, it was ultimately determined that a linear regression with a single transformed parameter fit the data better than a nonlinear model.

First, a single-factor regression analysis for each of the three parameters for each of the seven individual mixture designs across all six fines types was completed. This was done in order to observe trends across the similar mixture designs or fines types. Standard transformations listed in Table 10 were all attempted for all three parameters.

Table 10. Data transformations.

Transformation	
1	exp(x)
2	ln(x)
3	1/x
4	1/exp(x)
5	1/ln(x)
6	x^2
7	$1/x^2$

Several trends emerged across all seven mixture designs (regardless of percentage or type of cementitious replacement material). After the evaluation of each prediction variable using all transformations (and all combinations of the transformations given in Table 5), best fit transformed parameters were selected. The parameters which best fit the mixtures designs in single-factor linear regressions were exp(pH), conductivity and IR^2. This transformed data was then used for the remainder of the model analysis. This allowed for a linear regression analysis to be used rather than a nonlinear analysis. Each model contained only one transformed variable; therefore, the linear regression model with only one transformed variable produced a better fit than a nonlinear regression analysis, as most parameters fit well linearly.

Original pilot testing (Janssen 2010) indicated that an optimal fines replacement percentage might exist. The optimal fines replacement percentage was defined as a replacement percentage, which produced maximum performance. All data was analyzed for statistical significance based on Dunnett's testing to evaluate if a statistically optimal fines percentage existed. This testing revealed that an optimal fines percentage did not exist.

5.2 MODEL DEVELOPMENT

The six models were developed using the results from the laboratory testing. Two of the models predict set time, two predict 3-day compressive strength and two predict 28-day compressive strengths. A practitioner's model, which does not include particle size information, was developed for each of the three parameters. Additional, comprehensive models were then developed and require particle size information. The additional particle size information was found to strengthen models but this information is not readily available for the practical application of this work. The three models at each level predict the difference in set time, in minutes, from the control mixture, and both 3- and 28-day compressive strength, given as a percentage of the control mixture. For each model, the equation describing the prediction is given, as well as the coefficient of determination, R^2. The adjusted R^2 is also reported for each model, which accounts for possible size effects of the model (such that more predictor terms would produce a better fit, regardless of actual, significant relationship). The standard error of each model is also given. Finally, a plot of the measured values from the data set versus predicted values using the models are provided to display the fit of the model.

5.2.1 Practitioner's models

The first practitioner's model predicts the difference in set time, in minutes, from the control mixture. All data, including data which did not fulfill ASTM requirements, was used for the development of this model. The final regression equation is given as Equation 6.

$$\textit{difference in set time} = \frac{\left(11{,}066 - 0.648CaO - 6{,}250IR^2 - 0.018Cond\right) + 11.06}{0.61} \qquad (6)$$

Where,

CaO = percentage of CaO in the cementitious materials, including all supplementary cementitious materials

IR = index of refraction

Cond = Conductivity in μSiemens/cm

This model had an R^2 of 0.61 and an adjusted R^2 of 0.60 with a standard error of 18 minutes. The plot of measured versus predicted set time is given in Figure 28.

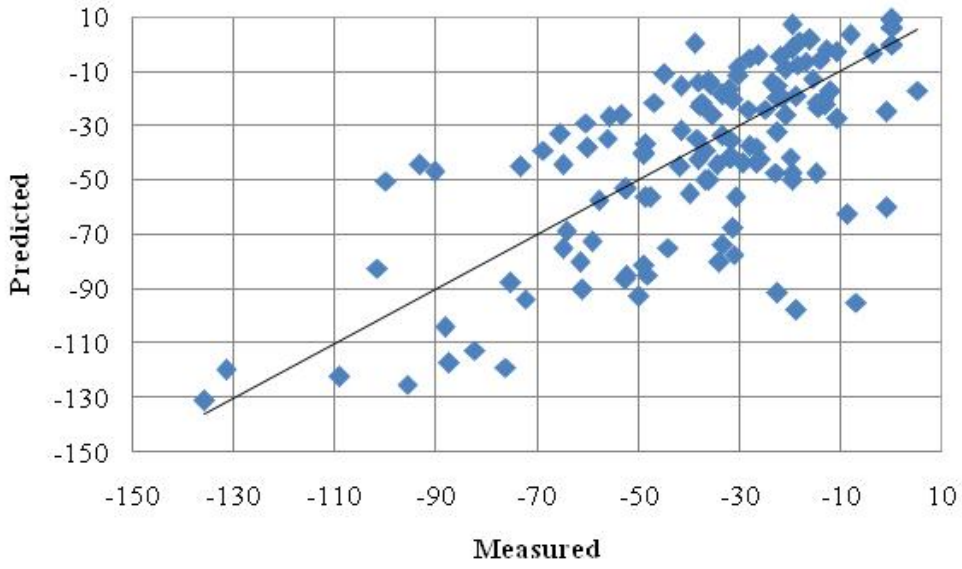


Figure 28. Measured versus predicted for the difference in set time practitioner's model.

The next practitioner's model predicts 3-day compressive strength as a percentage of the total control strength. The final regression equation for the second model is given as Equation 7.

$$3 \text{ day } \bar{f}_c \text{ percentage} = \frac{\left(-34.36 - 6.8\times10^{-5}\exp(pH) + 6.26\times10^{-3}Cond + 54.69CaORatio\right) - 23.81}{0.701} \quad (7)$$

Where,

pH = pH of the recycled water

Cond = Conductivity of the recycled water, measured in μSiemens/cm

CaO Ratio = ratio of CaO to SiO_2 and Al_2O_3 in the cementitious materials, as described in Equations 3 and 4, including all supplementary cementitious materials

This model had an R^2 of 0.76 and an adjusted R^2 of 0.75 with a standard error of 8.62%. The plot of measured versus predicted values is given in Figure 29.

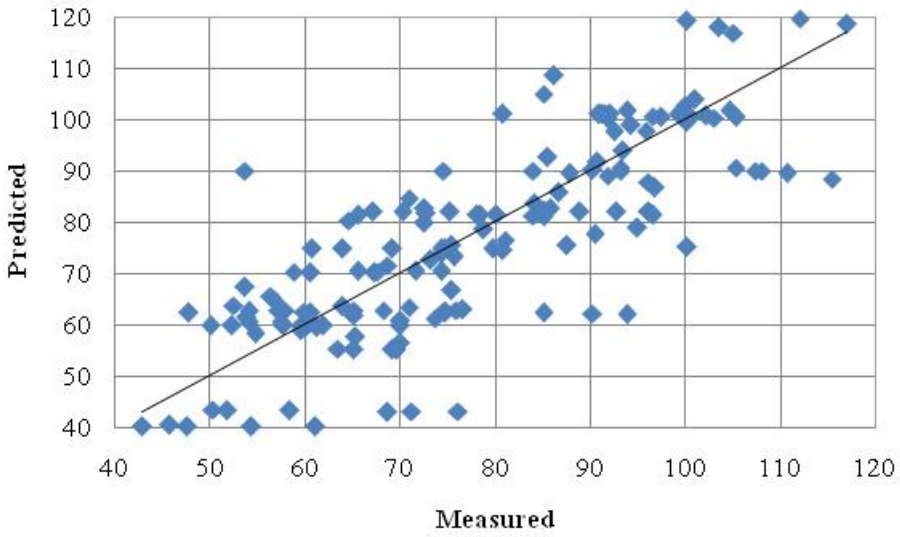


Figure 29. Measured versus predicted for the percentage of 3-day compressive strength practitioner's model.

The final practitioner's model predicts 28-day compressive strength as a percentage of the total control strength. The final regression equation for the second model is given as Equation 8.

$$28 \text{ day } \bar{f}_c \text{ percentage} = \frac{\left(24.41 - 2.5\times10^{-5}\exp(pH) - 0.00388Cond + 1.406CaO\right) - 52.69}{0.456} \quad (8)$$

Where,

pH = pH of the recycled water

Cond = Conductivity of the recycled water, measured in μSiemens/cm

CaO = percentage of CaO in the cementitious materials, including all supplementary cementitious materials

This model had an R^2 of 0.46 and an adjusted R^2 of 0.45 with a standard error of 8.24%. The plot of measured versus predicted values is given in Figure 30.

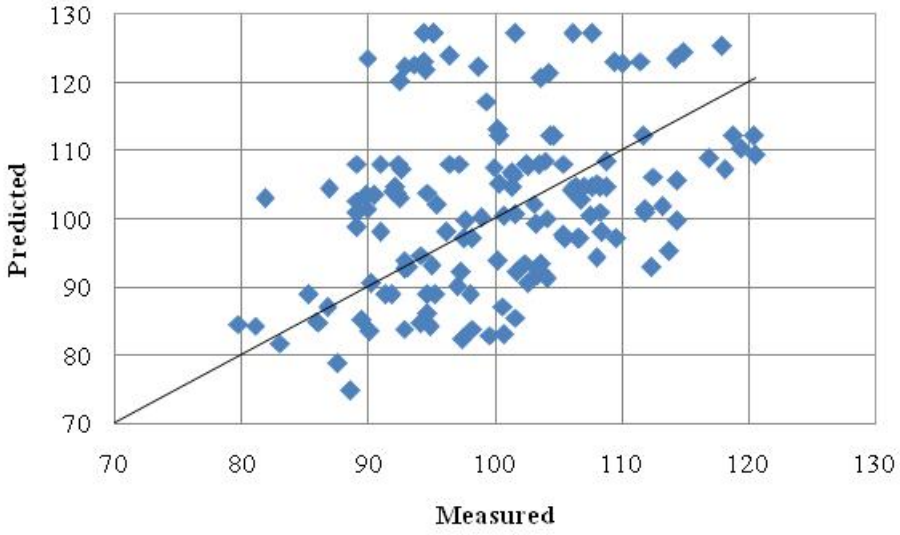


Figure 30. Measured versus predicted for the percentage of 28-day compressive strength practitioner's model.

5.2.2 Comprehensive models

The comprehensive models all require additional particle size information. The particle size distribution was previously calculated and given in Figure 10.

The first comprehensive model predicts the difference in set time, in minutes, from a control mixture. All data, including data which did not fulfill ASTM requirements, was used for the development of this model. The final regression equation is given as Equation 9.

difference in set time = (9)

$$\frac{\left(10016 - 10.52\left(d_{50}\right) + 0.6480CaO - 5656IR^2 - 0.00515Cond\right) + 9.411}{0.619}$$

Where,

d_{50} = the diameter based on the 50th percentile of the tested particles

CaO = percentage of CaO in the cementitious materials, including all supplementary cementitious materials

IR = index of refraction

This model had both an R^2 and an adjusted R^2 of 0.66 with a standard error of 17 minutes. The plot of measured versus predicted values is given in Figure 31.

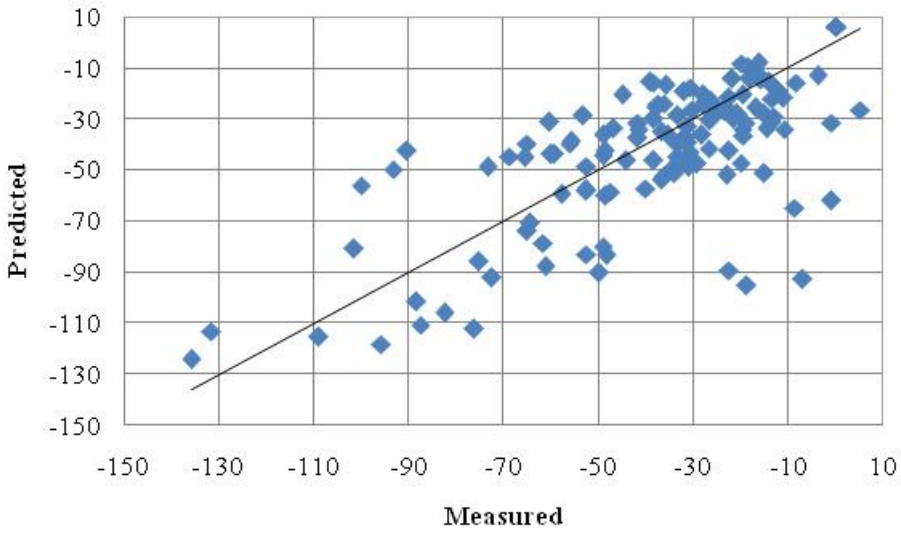


Figure 31. Measured versus predicted for the difference in set time comprehensive model.

The 3-day compressive strength as percentages of the control strength can be obtained using Equation 10 if the particle size characterization information is available.

3 day $\bar{f}_c$ percentage = (10)

$$\frac{\left(-35.95 - 7.7\times10^{-5} - 0.00227Cond + 54.63CaORatio + 20.2Span\right) - 23.29}{0.707}$$

Where,

Cond = Conductivity of the recycled water, measured in μSiemens/cm

CaO Ratio = ratio of CaO to Al_2O_3 and SiO_2 in the cementitious materials, including all supplementary cementitious.

Span = Boundary to describe the size distribution of the particles using the diameters of different percentiles of particles: d_{90}, d_{50}, and d_{10}.

This model had both an R^2 and an adjusted R^2 of 0.77 with a standard error of 8.57 %. The plot of measured versus predicted values is given in Figure 32.

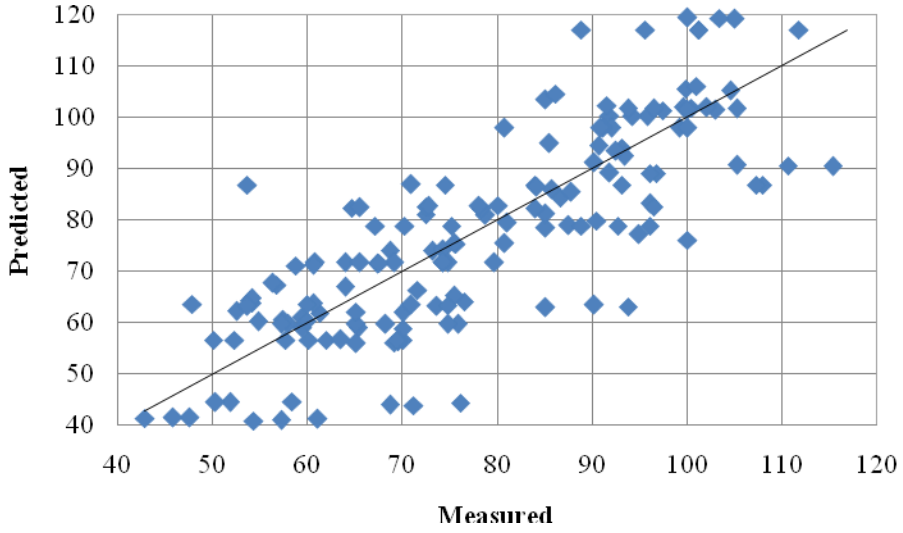


Figure 32. Measured versus predicted for the percentage of 3-day compressive strength comprehensive model.

The next two model predicts 28-day strength as percentages of the control strength. The final regression equation for is given as Equation 11.

$$28 \text{ day } \bar{f}_c \text{ percentage} = \frac{\left(22.6 - 0.0084Cond - 3.50\times10^{-5} + 1.406CaO + 22.87Span\right) - 50.65}{0.478} \quad (11)$$

Where,

pH = pH of the recycled water

Cond = Conductivity of the recycled water, measured in µSiemens/cm

CaO = percentage of CaO in the cementitious materials, including all supplementary cementitious materials

Span = Boundary to describe the size distribution of the particles using the diameters of different percentiles of particles: d_{90}, d_{50}, and d_{10} for the 90^{th}, 50^{th}, and 10^{th} percentiles, respectively

This model had an R^2 of 0.70 and an adjusted R^2 of 0.69 with a standard error of 6.2 %. The plot of measured versus predicted is given in Figure 33.

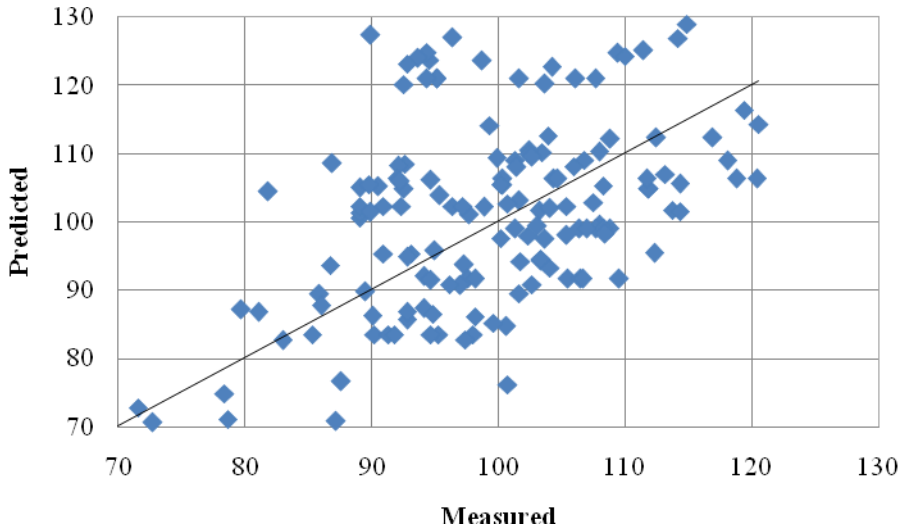


Figure 33. Measured versus predicted for the percentage of 28-day compressive strength comprehensive model.

All six models are summarized in Table 11 below, including the type, required input parameters, standard error and R^2.

Table 11. Summary of linear regression models.

Model type	Prediction	Input parameters	R^2	Adjusted R^2	Standard error
Practitioner's	Difference in set time	CaO % IR^2 Conductivity	61.0	60.3	18 minutes
Comprehensive		CaO % IR^2 Conductivity d_{50}	65.2	64.3	17 minutes
Practitioner's	Percentage of 3-day strength	exp(pH) Conductivity CaO Ratio	70.1	69.6	10.0%
Comprehensive		exp(pH) Conductivity CaO Ratio Span	70.9	70.1	9.9%
Practitioner's	Percentage of 28-day strength	exp(pH) Conductivity CaO %	45.7	44.7	8.5%
Comprehensive		exp(pH) Conductivity CaO % Span	47.8	46.5	8.4%

5.3 MODEL ADEQUACY

Linear regression modeling is structured on the assumption that the data fulfills certain criteria. There are five primary assumptions that must be verified: 1) that the relationship between the response and the regressors is approximately linear, 2) that the error term has a zero mean, 3) that the error term has a constant variance, 4) that the errors are uncorrelated and 5) that the errors are normally distributed (Montgomery et al 2012). Several procedures exist to test the normality assumptions of the data to ensure the validity of the linear regression models. These

include analyzing the residuals by examining plots and inspecting outliers, and inspecting potential multicollearity between predictors. Each of the six proposed models underwent model adequacy checking, which will be discussed in the following sections.

5.3.1 Residual analysis

Residuals in a linear regression analysis provide an approximation of the difference between the actual data and the fit of the model. This provides an estimate of the deviation of the model from the true data. Therefore, plotting residuals provides an effective visual means of investigating the model fit.

A normal probability plot ensures that the normality assumption of the regression assumptions is valid. A regression model with a cumulative normal distribution will plot as a straight line. Tailed or skewed plots, such that they deviate from linearity, indicate an inaccurate normality assumption. Finally, extreme residuals can indicate outlying observations, which would require further investigation. Normal probability plots are given for all six models in Figure 34.

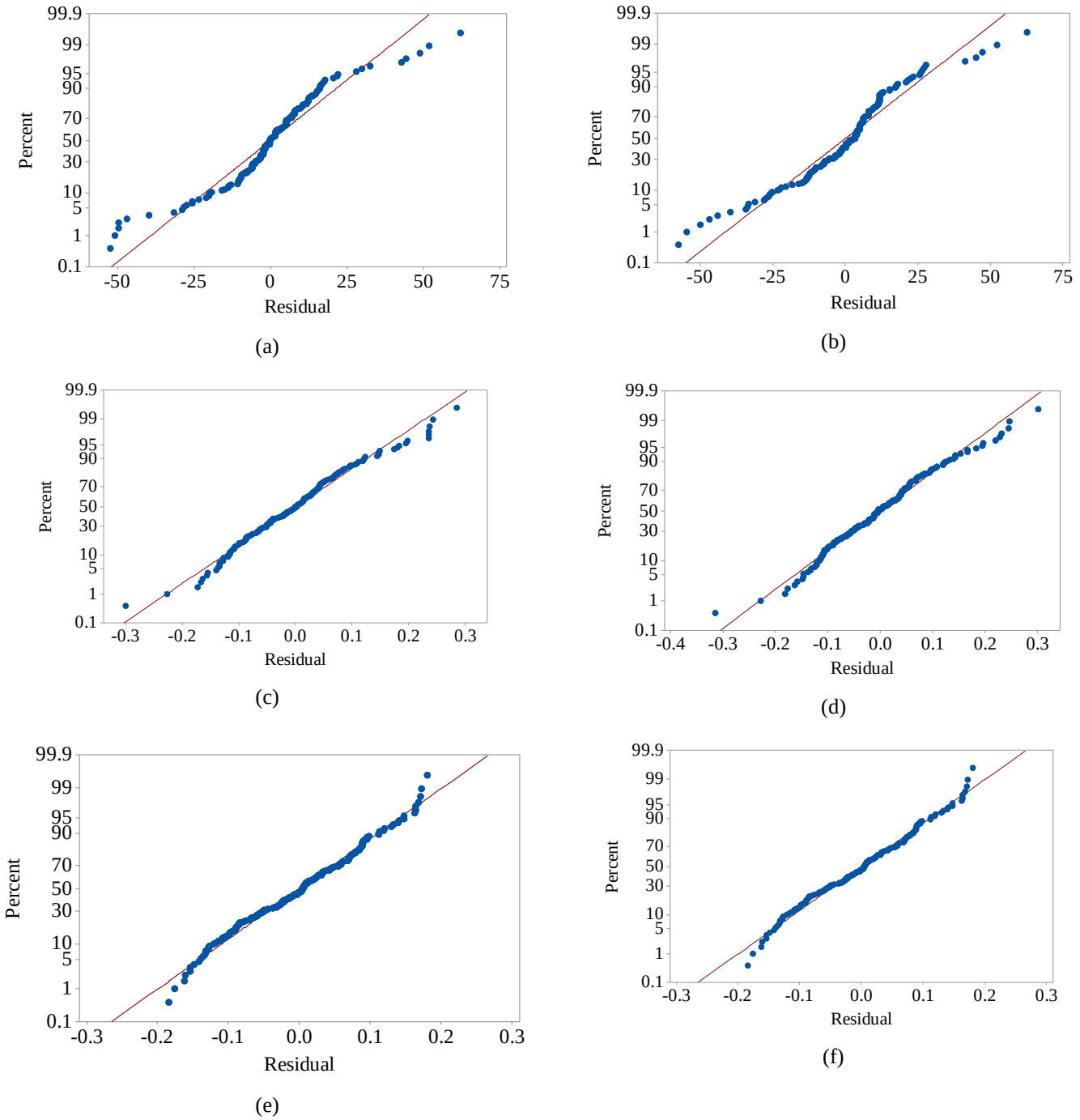


Figure 34. Normal probability plots for residuals for (a) comprehensive model for difference in set time (b) practitioner's model for difference in set time (c) comprehensive model for percentage of 3-day strength (d) practitioner's model for percentage of 3-day strength (e) comprehensive model for percentage of 28-day strength (f) practitioner's model for percentage of 28 day strength.

It can be seen from Figure 34 that all of the normality plots appear very linear. This ensures that the normality assumption is correct and valid for all six models.

Next, a plot of the residuals against the fitted values can be used to identify several types of model inadequacies such as non-constant variance and a lack-of-fit. The residuals should appear evenly distributed between two horizontal bands. Curved plots can indicate nonlinearity and funnel-shaped plots can indicate a problem with the variance assumption. A plot for all residuals against the fitted values for all six models is given in Figure 35.

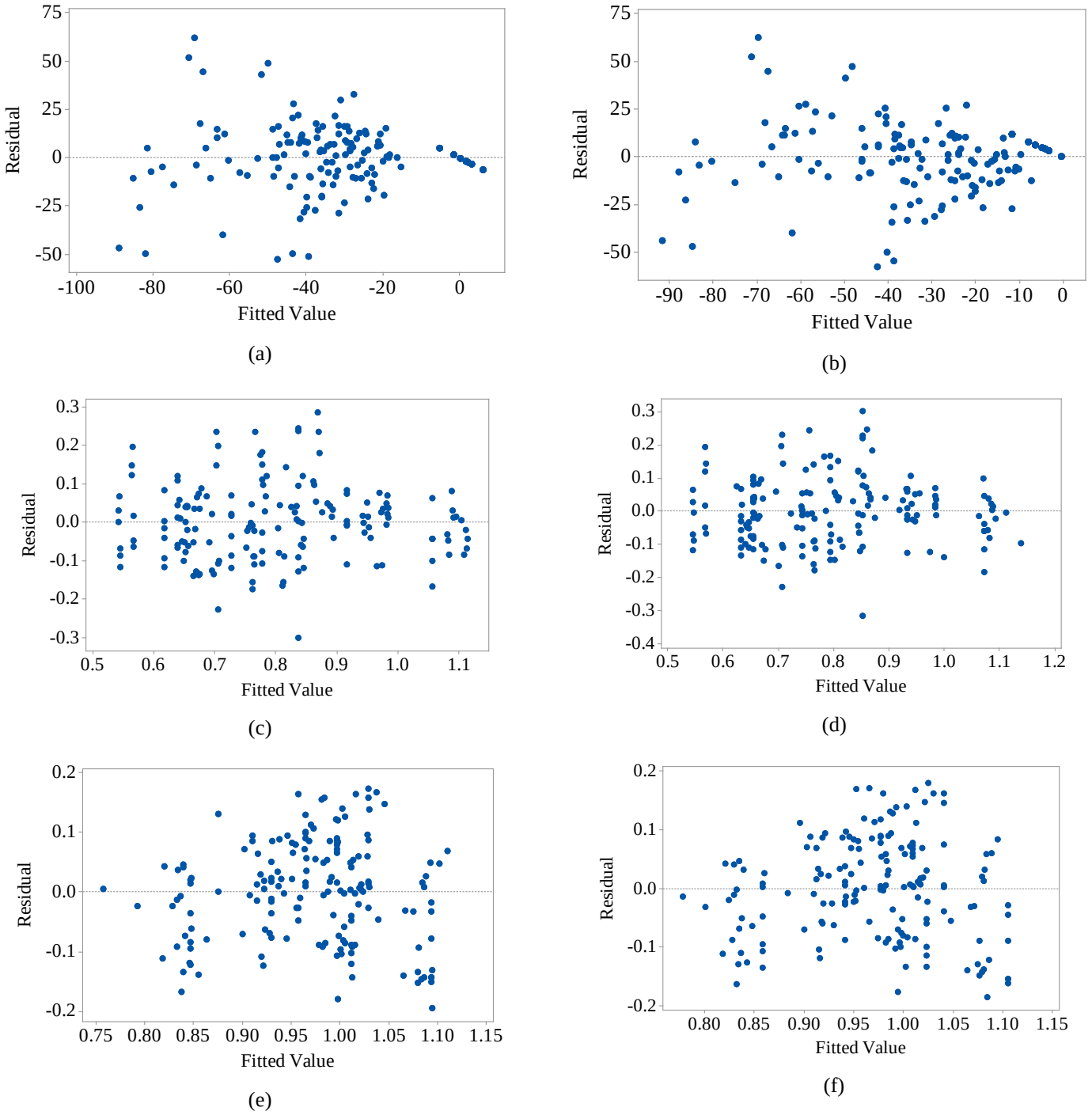


Figure 35. Residual versus fit plots for (a) comprehensive model for difference in set time (b) practitioner's model for difference in set time (c) comprehensive model for percentage of 3-day strength (d) practitioner's model for percentage of 3-day strength (e) comprehensive model for percentage of 28-day strength (f) practitioner's model for percentage of 28 day strength.

It can be seen from this figure that the residual versus fits plots appear to be well distributed between two horizontal bands, indicating adequate variance assumptions.

Residuals are assumed to be normally distributed with a mean of zero, an assumption that can be checked by histogram plots of the residuals. A normal distribution can be detected from these plots which would indicate an adequate residual assumption. Skews, tails, or otherwise deviations from normal distributions can be detected from these histograms as well. Histogram plots of all the residuals for each of the six models are given in Figure 36.

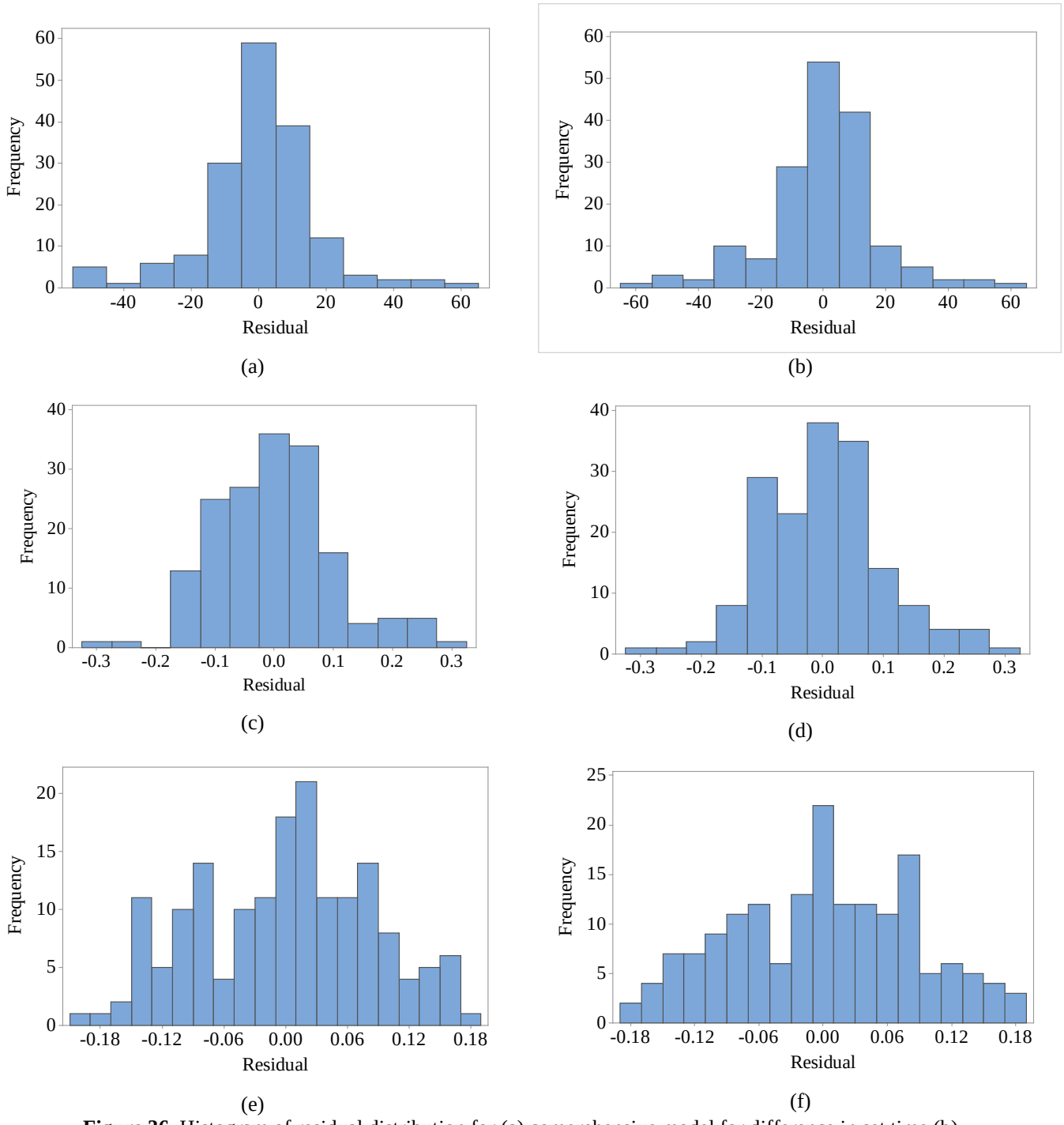


Figure 36. Histogram of residual distribution for (a) comprehensive model for difference in set time (b) practitioner's model for difference in set time (c) comprehensive model for percentage of 3-day strength (d) practitioner's model for percentage of 3-day strength (e) comprehensive model for percentage of 28-day strength (f) practitioner's model for percentage of 28 day strength.

All histogram plots appear to have an approximately normal distribution, indicating that the assumption that the distribution of residuals is normal and centered at zero is valid. Therefore, the summary of these plots has indicated that the normality assumptions are valid for these models.

5.3.2 Variance inflation factors

In addition to the plots from residual analysis, several other factors can be investigated in order to check the adequacy and stability of the linear regression models. First, variance inflation factors were investigated for each of the prediction variables in each regression equation. Variance inflation factors reveal important information regarding potential multicollinearity between the predictors. When predictors are multicollinear, the variance of the regression coefficients can become inflated, which is reflected by the variance inflation statistic. Generally, it is desired for a variance inflation factor to be less than five. The maximum variance inflation factor for each regression model is given in Table 12. It can be seen that none of the factors exceed five, indicating that multicollinearity between terms and therefore, model instability, is not a concern in any of the six models.

Table 12. Variance inflation factors.

Prediction model type	Prediction	Maximum variance inflation factor
Comprehensive	Percentage of 3-day strength	2.41
Practitioner's		1.09
Comprehensive	Percentage of 28-day strength	2.07
Practitioner's		1.00
Comprehensive	Difference in set time	2.00
Practitioner's		1.18

6.0 IMPLEMENTATION

6.1 MODEL VALIDATION

An established model, even a well-fitting model, is not guaranteed to fulfill its intended function. Therefore, model validation is necessary. There are several limitations to the model that was developed. First, it was developed only using mortar cube compression strength data rather than full concrete cylinders. Many tests were run and cost and time efficiency dictated casting small mortar cubes since it was assumed they would provide a close approximation to the performance of concrete. The final intention of the model, however, is to predict the behavior of concrete rather than mortar. Additionally, the regression model used material characterization data from multiple recycled fines sources under extremely controlled conditions, where the fines sources were dried, sieved, mixed, and measured carefully by mass. In reality, however, these fines sources will be included as waste water with only the three in-line measurements to characterize the material properties. These differences can potentially have an impact on the prediction capabilities of the regression model and therefore model validation is necessary.

Generally, three validation techniques can be used: 1) analysis of the model coefficients by comparing with experience, theory, or simulation; 2) collection of new data and 3) data splitting.

For this specific data set, option 1 was not feasible as there is extremely limited, scattered and unreliable previous work and no simulations were run. Option 3 was possible, but given the unknown behavior of this experiment, it seemed a more robust model would be possible if all data was included. Therefore, the regression model will be validated using option 2 of collecting new data.

6.2 MOCKUP WATER SUPPLY SYSTEM

In order to validate the model, a mockup water supply system simulating those typically used in a batch plant was used to make concrete. Again, despite the fact that the initial model was developed for mortar samples only, the ultimate application is concrete performance prediction. A water supply system was instrumented with in-line sensors for monitoring pH, conductivity, and percent solids as shown in Figure 37. The sensor output devices are shown in Figure 38.

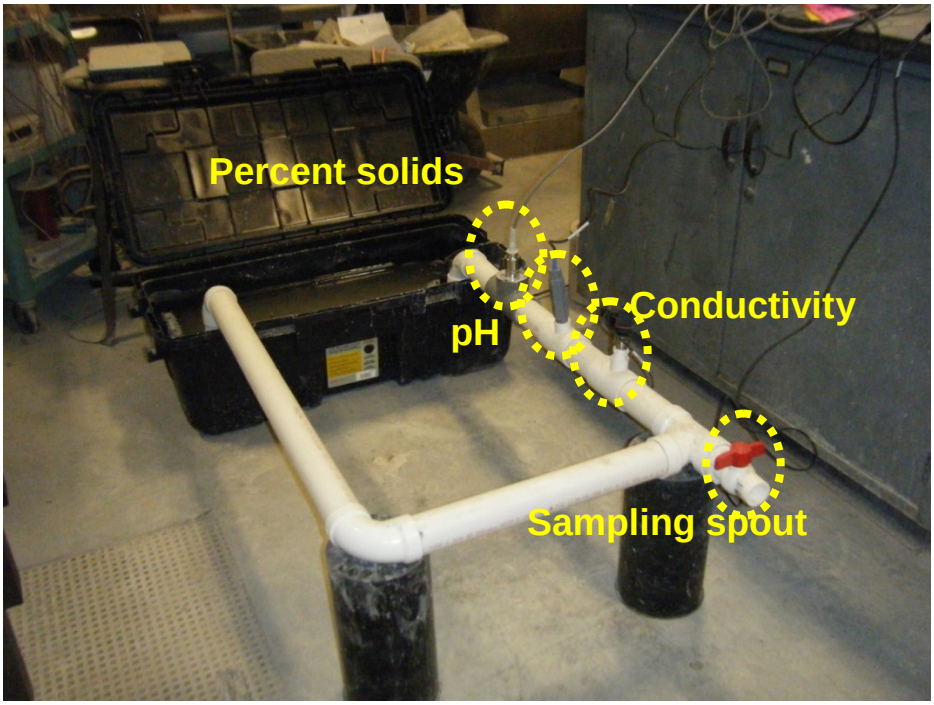


Figure 37. Water recirculation system with in-line sensors.

Figure 38. Sensor output devices.

The sensors used in this laboratory study along with their sensitivities and accuracies are given in Table 13.

Table 13. Equipment accuracy and resolution for water recirculation system.

Property measured	Accuracy	Resolution
Percent solids	±5% of range 0.0006 (for IR)	0.01%
Conductivity	±1%	1 µS/cm
pH	0.01	0.01

It is important to note that the equipment used to measure IR was a percent solids meter, which is converted into IR. The percent solids meter was deemed to be more appropriate for this application. In order to convert between percent solids and index of refraction, an initial material characterization using the original six types of fines was completed with the percent solids meter. Then, a linear regression between the two measurements was performed in order to convert between the two types of measurements. A plot of the materials characterization of the six fines types is given in Figure 39.

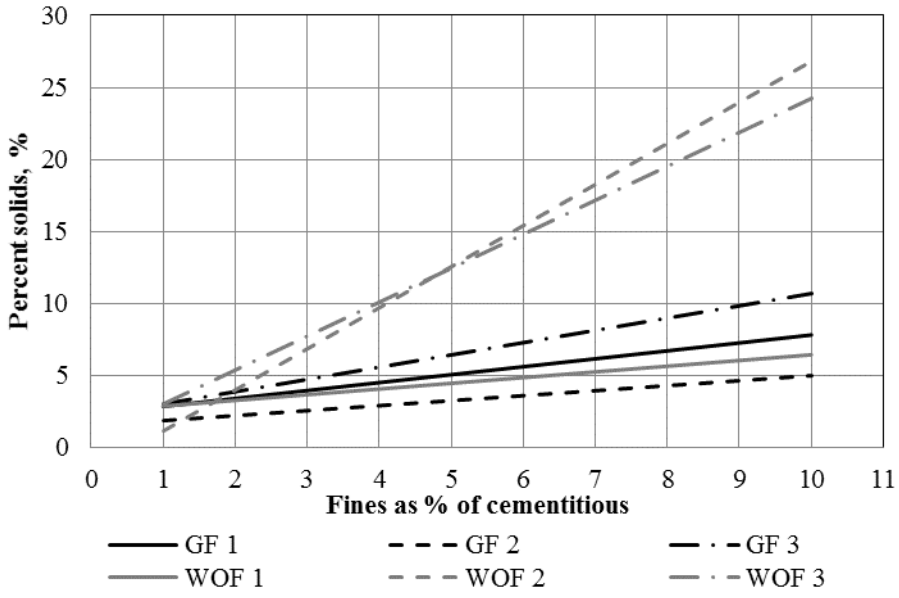


Figure 39. Particle content as a function of fines concentration.

The linear regression relationship developed for predicting IR based on the particle content is described by Equation 13 given below.

$$IR = 1.33289 + 0.00019857PS \quad (13)$$

Where,

PS = percent solids, %

This relationship had an R^2 of 0.92. The standard error for the estimate was 0.000312.

6.2.1 Results

In order to validate the predictive models, three concrete mixtures were cast: one control and two batches with different waste water. All three concrete mixtures had a supplementary cementitious replacement material of 15% Class F fly ash. The same fly ash from the original mortar testing was used. From each batch, slump, set time, and 3-day and 28-day compressive strength testing was completed. Material properties are given for the coarse and fine aggregate, and the cement and fly ash in Table 14.

Table 14. Detail of materials used in validation concrete mixtures.

Coarse aggregate	
Type	River gravel
Top size	1.0 in
Bulk specific gravity (SSD)	2.50
Absorption capacity	2.07 %
Los Angeles abrasion value	34%
Fine aggregate	
Fineness modulus	2.86
Absorption capacity	1.24%
Bulk specific gravity (SSD)	2.62
Cementitious materials	
Cement type	ASTM Type I Portland
Fly ash type	Class F

The mixture design for all three mixtures was then constructed based on the concrete mixture requirements outlined in Table 15. The final mixture design is provided in Table 16.

Table 15. Mix design criteria used to design concrete mixtures.

Criteria	Value
w/cm ratio	0.42
Slump, in	4
Min. 28-day compressive strength	4,500 psi
Target 28-day compressive strength	5,500 psi
Min. cement requirements	520 lb/CY
Approximate air content (not entrained)	1.5%

Table 16. Concrete mixture proportions.

Component	Proportion (lbs/cyd)
Cement	587
Fly ash	104
Fine aggregate	1,218
Coarse aggregate	1,816
Water	281

Concrete was mixed in a standing drum mixer in accordance with ASTM C 192: Practice for Making and Curing Concrete Test Specimens in the Laboratory. Slump was then measured for each mixture and was tested according to ASTM C 143: Test Method for Slump of Hydraulic-Cement Concrete. Following slump testing, cylinders were cast according to ASTM C 39: Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens. A total of 8 cylinders were cast with four allocated towards 3-day and four for 28-day compressive strength measurements. Finally, mortar was separated from the concrete mixture (by sieving the coarse aggregate out of the concrete with a No. 4 screen) for performing the Vicat testing of mortar samples. It was determined that even though penetration testing is the standard for concrete testing, the Vicat testing would better simulate the predicted models since Vicat test data was used in the model development.

Two of the three concrete mixtures contained recycled waste water as the mix water in the concrete. Randomized mixtures of dried fines were blended into the water in order to make a completely new waste water mixture. The mixture was intended to be a completely unknown, randomized mixture in order to simulate conditions that would be experienced in an in-line water supply system in a ready-mix concrete plant. Therefore, only the readings taken from the in-line sensors were used and no quantification of the behavior of the fines was otherwise considered. Readings from the three in-line sensors were then taken while the water was being pumped through the system, such that sufficient agitation was present to keep the solids suspended and thoroughly mixed in the water. Water was then pulled from the sampling spout and used as mixing water for making the concrete. The in-line sensor measurements for the water used in the concrete are provided in Table 17.

Table 17. In-line sensor measurements for two validation mixtures.

Concrete mixture	pH	Conductivity, μSiemens/cm	Percent solids	IR[1]
Mixture 1	11.27	1184	8.75	1.33465311
Mixture 2	11.14	680	14.3	1.33603868

[1]IR was calculated from the percent solids using Equation 13

The results from this testing for all three batches are given in Table 18. Fresh water without the addition of recycled fines was used for the control mix.

Table 18. Measured raw data for the two validation mixtures.

Concrete mixture	Slump, in	Set time, minutes	3-day compressive strength, psi	28-day compressive strength, psi
Control	5	210	4020	6060
Mixture 1	3	229	4280	6370
Mixture 2	3	251	4390	6210

6.2.2 Predictions

To validate the models, the data from the testing described in the previous section will now be used with the practitioner's predictive models given in Equations 6-8. First, the raw data given in Table 17 was converted into the prediction values: difference in set time, and the percentages of strength with respect to a control and are provided in Table 18. It can be seen from the results in Table 18 that both mixtures fulfilled the requirements outlined in ASTM C 1602 for mixing water in fresh concrete production. Both 3-day and 28-day compressive strengths met and exceeded the requirement since at least 90% of the compression strength of the control mixture was achieved. Likewise, the difference in set time from the control mixture did not exceed the 60 minute threshold outlined in the specification. Both mixtures fell well within these limitations despite containing a percent solids value (from Table 16) that far exceeded the

5% set as a limitation in the specification. The practitioner's models (Equations 6 through 8) were then used to calculate the predictions also given in Table 19. The standard errors calculated for each prediction model are given in parenthesis next to the corresponding prediction model.

Table 19. Measured and predicted concrete properties.

Concrete mixture	Difference in set time, minutes	Percentage of 3-day compressive strength	Percentage of 28-day compressive strength
Mixture 1- Measured	-19	106	105
Mixture 1- Predicted	-71(18.0)	91 (8.62)	87 (8.24)
Mixture 2- Measured	-41	109	102
Mixture 2- Predicted	-93 (18.0)	88 (8.62)	91 (8.24)

These results indicate several discrepancies with the prediction equations. First, the difference in set time, was not close to the intended value. However, the difference between the two measured values was close to the difference for the predicted values for the two batches. The predicted range, based on the standard error of the prediction equation, exceeds the measured difference between the two predictions indicating that a problem may exist between the absolute accuracy of the model rather than the relative accuracy. Additionally, this accuracy discrepancy could be attributed to using the Vicat testing apparatus for characterizing set time of the mortar extracted from the concrete rather than using the standard penetration testing device. The predictions for percentage of 3-day compressive strengths indicate close predictions. However, the percentage of 28-day strength does not indicate a close prediction. This model had the worst fit of the three separate parameters being modeled and therefore the prediction, even with the mortar cubes, was not very accurate. The predictions for both of the 28-day strength mixtures far exceeded the predicted value plus its standard error. In both cases, however, the measured strength exceeded the predicted strength and exceeded the limit of the percentage of solids in the waste water established by the ASTM C 1602 specification.

Despite these initial reasons explaining the discrepancy between the predicted and measured values, several other factors may contribute to these differences. First, it must be noted that all prediction models were built using mortar strength data. Fortunately, the prediction is based on difference in strength from a control rather than absolute strength, but it should be noted that concrete and mortar strength are inherently different. ASTM C 109, which specifies the testing procedure for mortar cube strength, states, “Caution must be exercised in using the results of this test method to predict the strength of concretes.” Similarly, the mixture design for the concrete contained a higher sand to cement ratio than the mortar mixture. This would decrease the overall set time and possibly affect the other parameters as well.

Other discrepancies between the concrete behavior and mortar behavior could be attributed to the inclusion of coarse aggregates in the concrete mixtures (a smooth river gravel was used for all mixtures) and a river sand was used for the concrete mixtures, whereas a much more uniformly graded Ottawa sand was used for the mortar mixtures.

However, the most pronounced difference in results can most likely be attributed to the use of the fines in the waste water as a replacement material during the prediction of the models. The fines were initially treated as a replacement for the cementitious materials. However, the waste water as a whole was used in the concrete mixture strictly as water and not as a replacement for cementitious materials. This different treatment of the recycled fines probably has the greatest effect on the discrepancy between the predicted results and the measured results.

7.0 USER GUIDELINES

The prediction models show that measurements of several key parameters can be used in order to predict performance. These prediction models can be used to predict concrete behavior based on the in-line measurements taken from the waste water. In order to provide guidelines for the use of these prediction models, plots were created to present the sensitivity of the basic level predictions to specific parameters. Because the final output is a combination of three input variables, the output can vary greatly based on the combination of these parameters. Plots describing the relationship between the predicted percentage of 3-day compression strength and the conductivity measured in μSiemens/cm are given below. Four plots are presented in Figure 40 to Figure 43 with pH levels of 9, 10, 11, and 12, respectively. Five different curves are plotted on each graph for different levels of the CaO ratio, which helps account for the effects of supplementary cementitious materials.

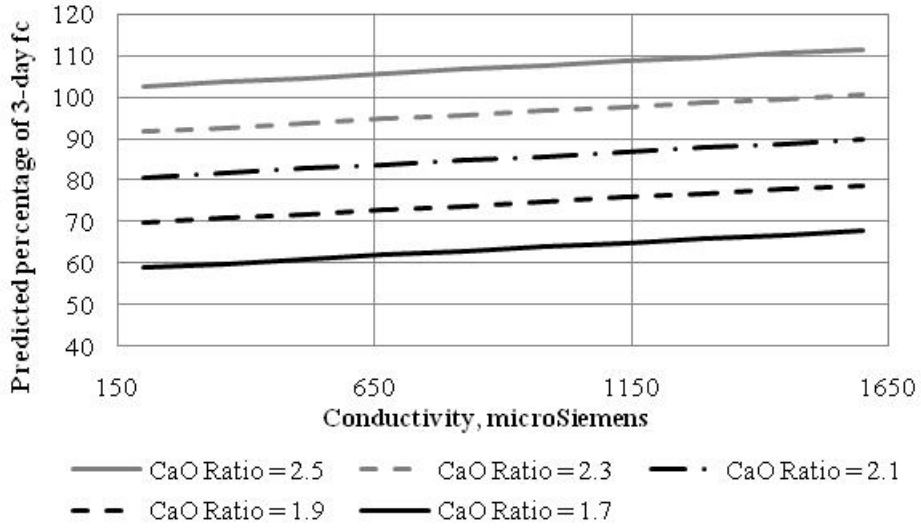


Figure 40. Predicted percentage of 3-day strength vs. conductivity for pH = 9.

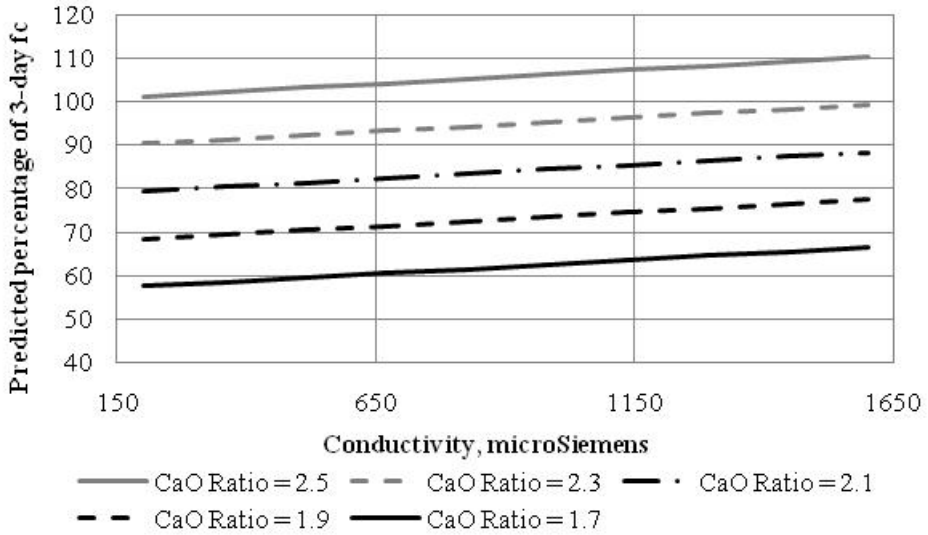


Figure 41. Predicted percentage of 3-day strength vs. conductivity for pH = 10.

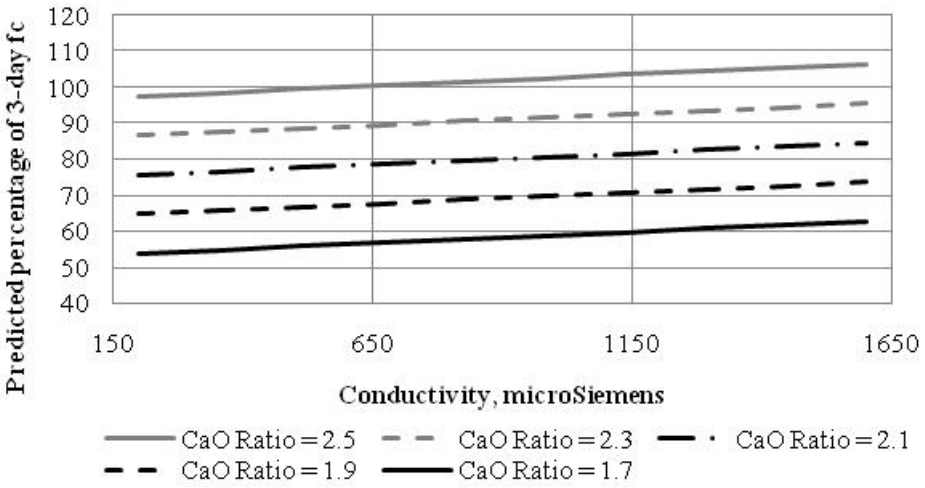


Figure 42. Predicted percentage of 3-day strength vs. conductivity for pH = 11.

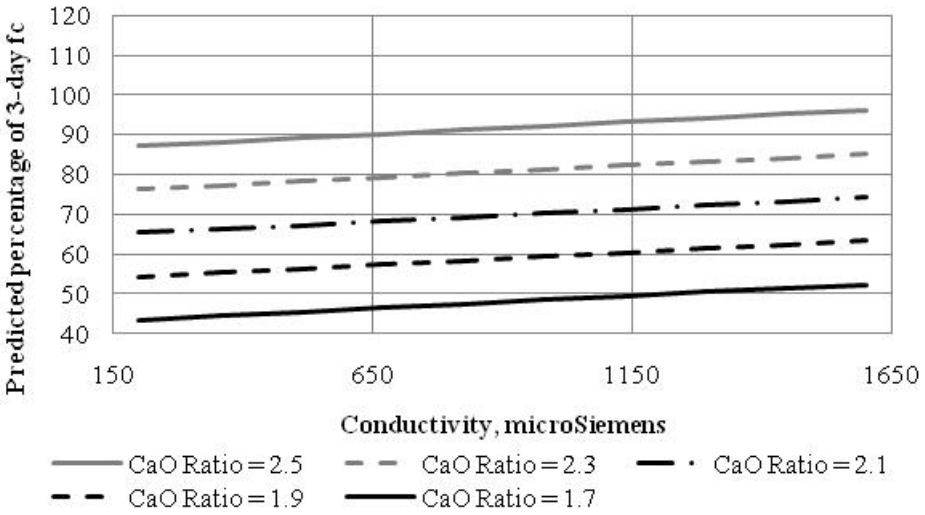


Figure 43. Predicted percentage of 3-day strength vs. conductivity for pH = 12.

Plots describing the relationship between the predicted percentage of 28-day compressive strength and the conductivity measured in μSiemens/cm are given below. Four plots are presented from Figure 44 to Figure 47 with pH levels of 9, 10, 11, and 12, respectively. Five different curves are plotted on each graph representing different levels of CaO.

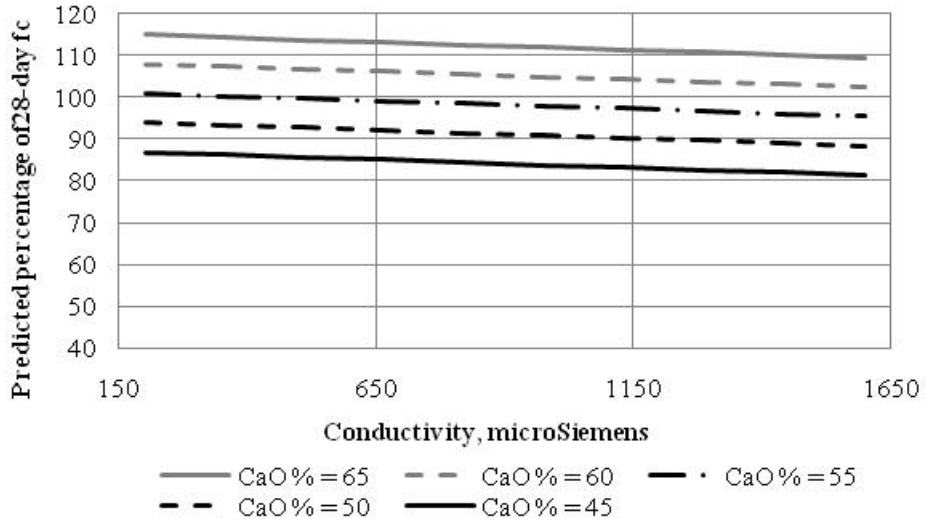


Figure 44. Predicted percentage of 28-day strength vs. conductivity for pH = 9.

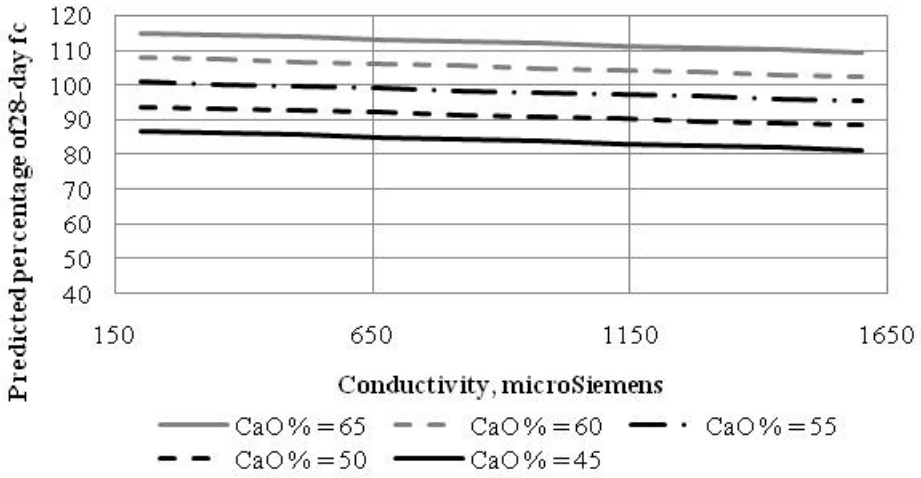


Figure 45. Predicted percentage of 28-day strength vs. conductivity for pH = 10**.**

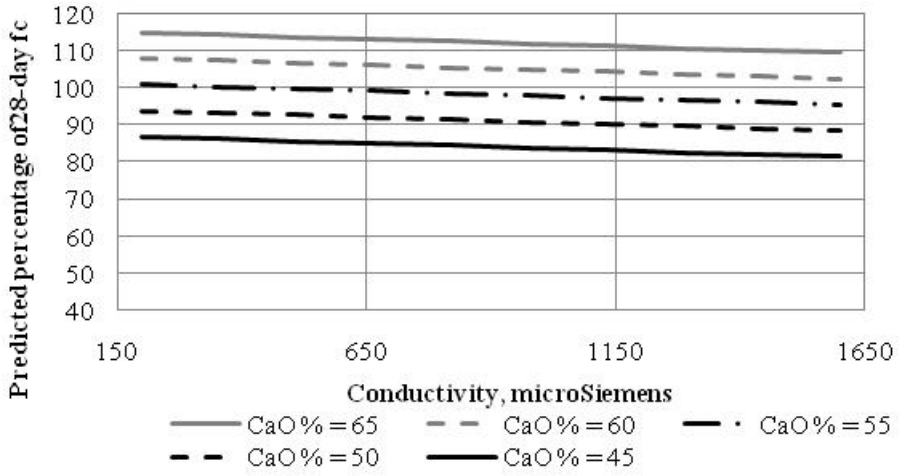


Figure 46. Predicted percentage of 28-day strength vs. conductivity for pH = 11.

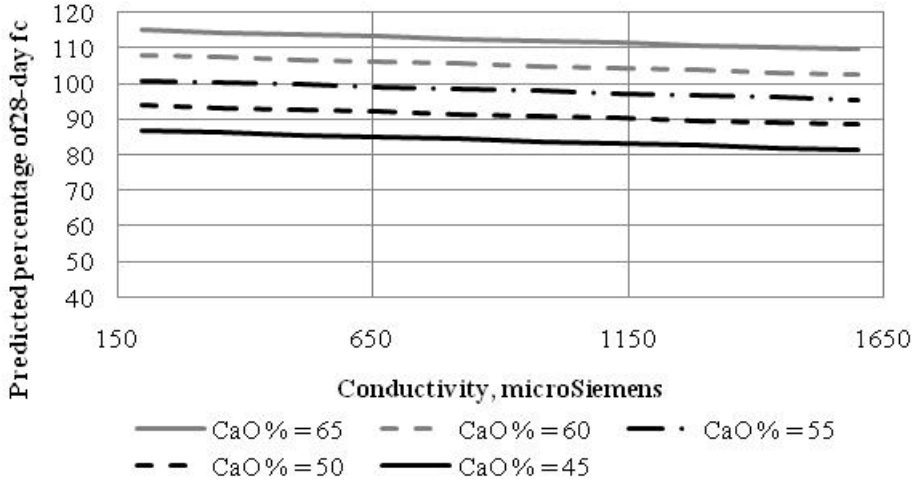


Figure 47. Predicted percentage of 28-day strength vs. conductivity for pH = 12.

Plots which describe the relationship between the predicted difference in set time and the index of refraction are given below. Four plots are presented from Figure 48 to Figure 51 with different conductivity levels of 200, 500, 1000, and 1500 μSiemens/cm, respectively. Five different curves are plotted on each graph representing different levels of CaO.

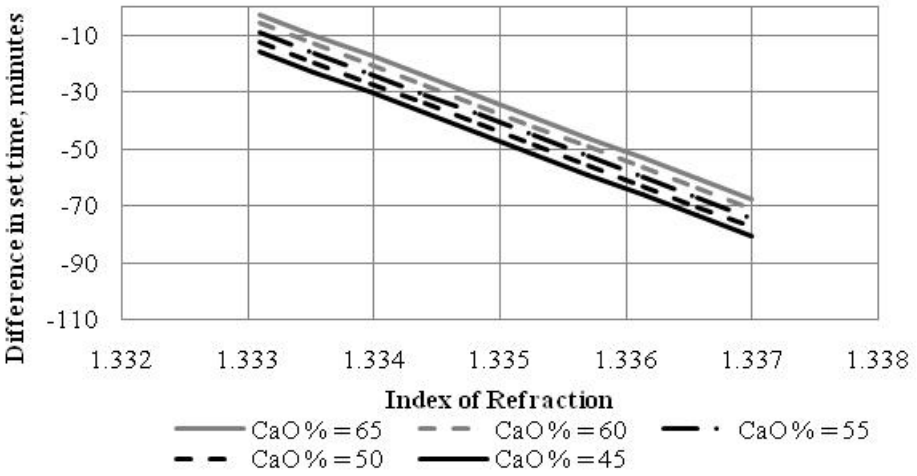


Figure 48. Predicted difference in set time vs. IR for conductivity = 200 μSiemens/cm.

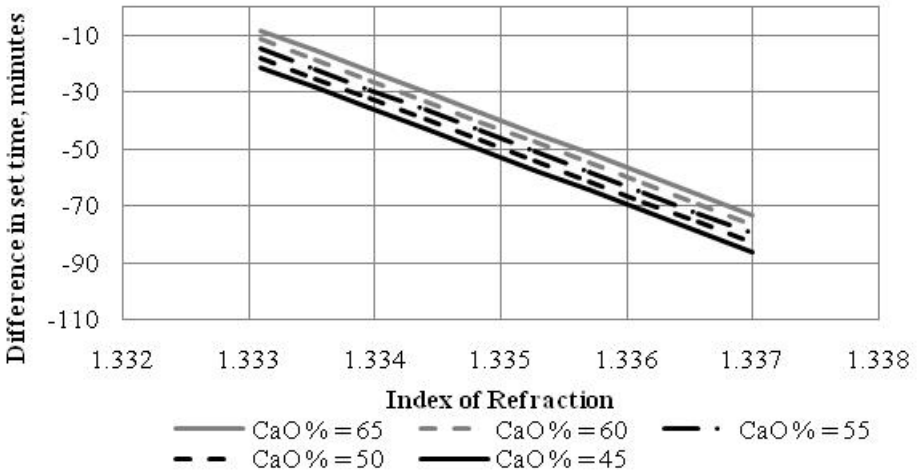


Figure 49. Predicted difference in set time vs. IR for conductivity = 500 μSiemens/cm.

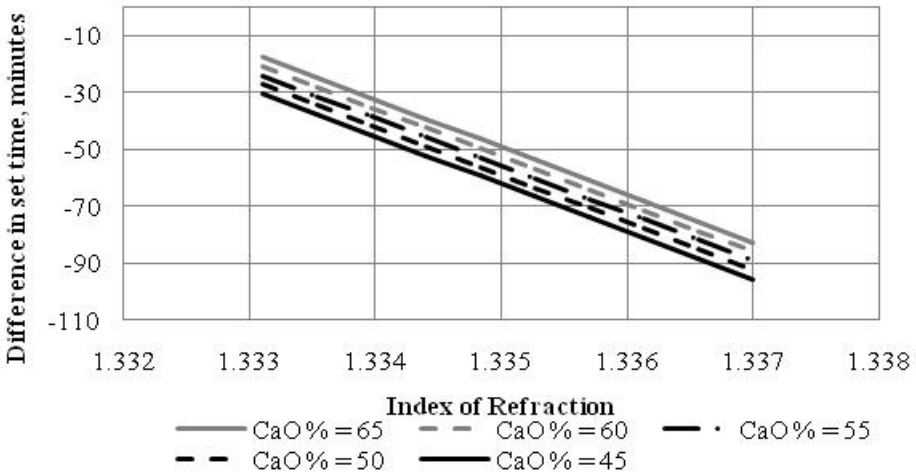


Figure 50. Predicted difference in set time vs. IR for conductivity = 1000 μSiemens/cm.

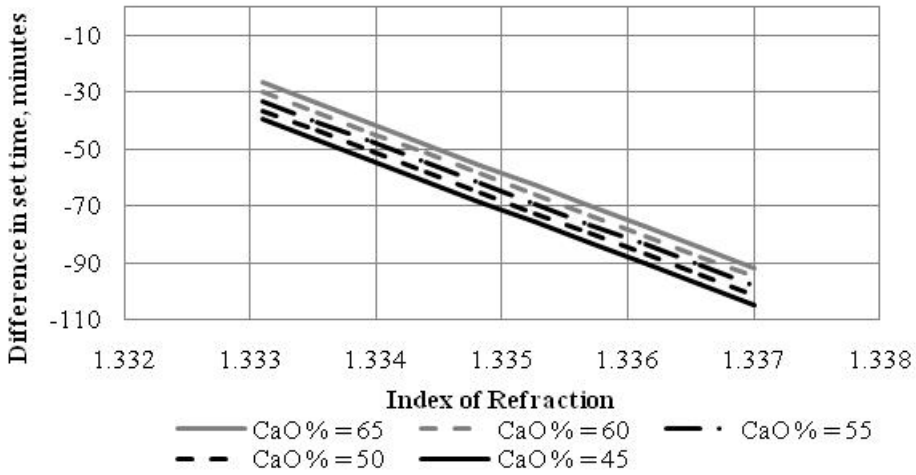


Figure 51. Predicted difference in set time vs. IR for conductivity = 1500 μSiemens/cm.

These plots provide a resource to users of the prediction models to assist in visualizing the relationship between the relevant variables in each equation and to provide insight into the performance of the mixture.

8.0 CONCLUSIONS

The waste water from a variety of sources, including grinding operations and ready mix truck wash out, can be characterized through several key parameters in order to predict set time and compression strength, as required in ASTM C1602. Concrete mix water containing a higher solids content than allowed under ASTM C1602 might be suitable for use in new concrete. The hydrated and unhydrated cement particles can serve as nucleation sites, thus expediting the hydration reaction. Improved particle packing is another positive effect that can be achieved through the presence of the cement particles in the waste water. Characterization of the waste water for use requires additional parameters, along with those traditionally used, such as IR, specific gravity, or fines content.. This work has shown that a combination of conductivity, IR, and CaO are sufficient for water characterization in order to predict the performance parameters of a concrete mixture.

Six predictive models were developed based on mortar testing in order to predict the difference in set time from a control mixture, and the 3-day and 28-day compression strengths as a percentage of the control strength of the mixture. These three models were computed over two separate levels: 1. a practioner's level, which does not include particle size information and therefore is more applicable for immediate implementation in a ready-mix concrete plant, and 2. a comprehensive level, which includes particle size information and ultimately produced more accurate models. Finally, a mock-up water supply system was constructed in the lab to be used in making concrete. Comparisons were then made between the predicted values, based on equations developed with the mortar test results, to that of actual concrete samples. The agreement between the

performance of the concrete samples and the prediction models varied, but sufficient evidence was provided to validate the concept and provide guidance on the direction of future work needed to further refine the process.

Future work needed includes the development of a database for concrete mixtures, similar to the database developed for mortar mixtures. Clearly, concrete mixtures behave differently when using waste water as a replacement for fresh water than the mortar mixtures, which incorporated the waste water as a replacement of the total cementitious materials. The implementation of a full scale system in a concrete ready mix plant, including the monitoring of the in-line sensor readings and the resulting concrete performance, would be helpful in populating a database of concrete mixture-performance information. This would allow for further exploration of the relationships between the recycled water and full-scale concrete production.

APPENDIX A

SUPPLEMENTARY CEMENTITIOUS MATERIAL MILL SHEETS

LAFARGE CEMENT **Cement Test Report**

Mill Test Report Number:	SEA_NEWCEM_JAN12
YEAR:	2012
MONTH:	February
PLANT:	Seattle
CEMENT TYPE:	Grade 100 NewCem

Reference Cement

Fineness by Air Permeability (m^2/kg; ASTM C204)	414	
Fineness by 45 µm (No. 325) Sieve (% retain; ASTM C430)	3.3	
Compressive Strength (ASTM C109/C109 M)	psi	
7-day	4,400	
28-day	5,520	
	Actual	Max Limit
Total Alkalies ($Na_2O + 0.658\ K_2O$) (%, ASTM C114)	0.85	0.9

Slag

CHEMICAL ANALYSIS	Percent
Silica Dioxide (SiO_2; ASTM C114)	31.3
Ferric Oxide (Fe_2O_3; ASTM C114)	0.8
Aluminum Oxide (Al_2O_3; ASTM C114)	13.1
Calcium Oxide (CaO; ASTM C114)	43.6
Sulfur Trioxide (SO_3; ASTM C114)	4.7
Magnesium Oxide (MgO; ASTM C114)	3.7
Potassium Oxide (K_2O; ASTM C114)	0.5
Titanium Oxide (TiO_2, ASTM C114)	0.5
Loss on Ignition (L.O.I.; ASTM C114)	2.3
Inorganic Process Addition	6

Slag

Fineness by Air Permeability (m^2/kg; ASTM C204)	472		
Fineness by 45 µm (No. 325) Sieve (% retain; ASTM C430)	3.7		
Compressive Strength (ASTM C109/C109 M)	psi	SAI	SAI Limit Min
7-day	3,670	83	75
28-day	6,580	119	95
Specific Gravity (Mg/m^3; ASTM C188)	2.87		
	Actual	Max Limit	
Air Content of Mortar (%, ASTM C185)	5.3	12	
Sulfide Sulfur (% S, ASTM C114)	0.7	2.5	
Sulfate Ion (% as SO3, ASTM C114)	3.0	4	

The ground granulated blast furnace slag complies with the current specification of the chemical physical requirement of ASTM C-989, AASHTO M-302 for grade 100 Ground Granulated Blast Furace Slag (GGBFS) and and CSA A3001 Slag.

Certified by:

Daniel Waldron
Quality Control Laboratory Supervisor
February 16, 2012

Figure 52. Mill testing information for slag used in laboratory.

HEADWATERS RESOURCES

Adding Value to Energy ™

ASTM C618 / AASHTO M295 Testing of
Hatfield Ferry Fly Ash

Sample Type: 3200-ton **Report Date:** 8/22/2012

Sample Date: 5/15 - 5/23/12 **MTRF ID:** 1383HF

Sample ID:

Chemical Analysis			ASTM / AASHTO Limits Class F	Class C	ASTM Test Method
Silicon Dioxide (SiO2)	48.72	%			
Aluminum Oxide (Al2O3)	23.03	%			
Iron Oxide (Fe2O3)	18.81	%			
Sum of Constituents	90.56	%	70.0% min	50.0% min	D4326
Sulfur Trioxide (SO3)	0.54	%	5.0% max	5.0% max	D4326
Calcium Oxide (CaO)	3.42	%			D4326
Moisture	0.11	%	3.0% max	3.0% max	C311
Loss on Ignition	1.90	%	6.0% max 5.0% max	6.0% max 5.0% max	C311 AASHTO M295
Available Alkalies, as Na2O **When required by purchaser**	0.61	%	not required 1.5% max	 1.5% max	C311 AASHTO M295
Physical Analysis					
Fineness, % retained on #325	21.43	%	34% max	34% max	C311, C430
Fineness Uniformity	2.22	%	5% max	5% max	
Strength Activity Index - 7 or 28 day requirement					C311, C109
7 day, % of control	80	%	75% min	75% min	
28 day, % of control	84	%	75% min	75% min	
Water Requirement, % control	101	%	105% max	105% max	
Autoclave Soundness	-0.01	%	0.8% max	0.8% max	C311, C151
Density	2.53				C604
Density Uniformity	0.59	%	5% max	5% max	

Headwaters Resources certifies that pursuant to current ASTM C618 protocol for testing, the test data listed herein was generated by applicable ASTM methods and meets the requirements of ASTM C618 for Class F fly ash.

Bobby Bergman
MTRF Manager

AAR AASHTO R18

Materials Testing & Research Facility
2650 Old State Highway 113
Taylorsville, Georgia 30178
P: 770.684.0102
F: 770.684.5114

Figure 53. Mill testing information for Class F fly ash used in laboratory.

APPENDIX B

COMPLETE MATERIALS CHARACTERIZATION PLOTS

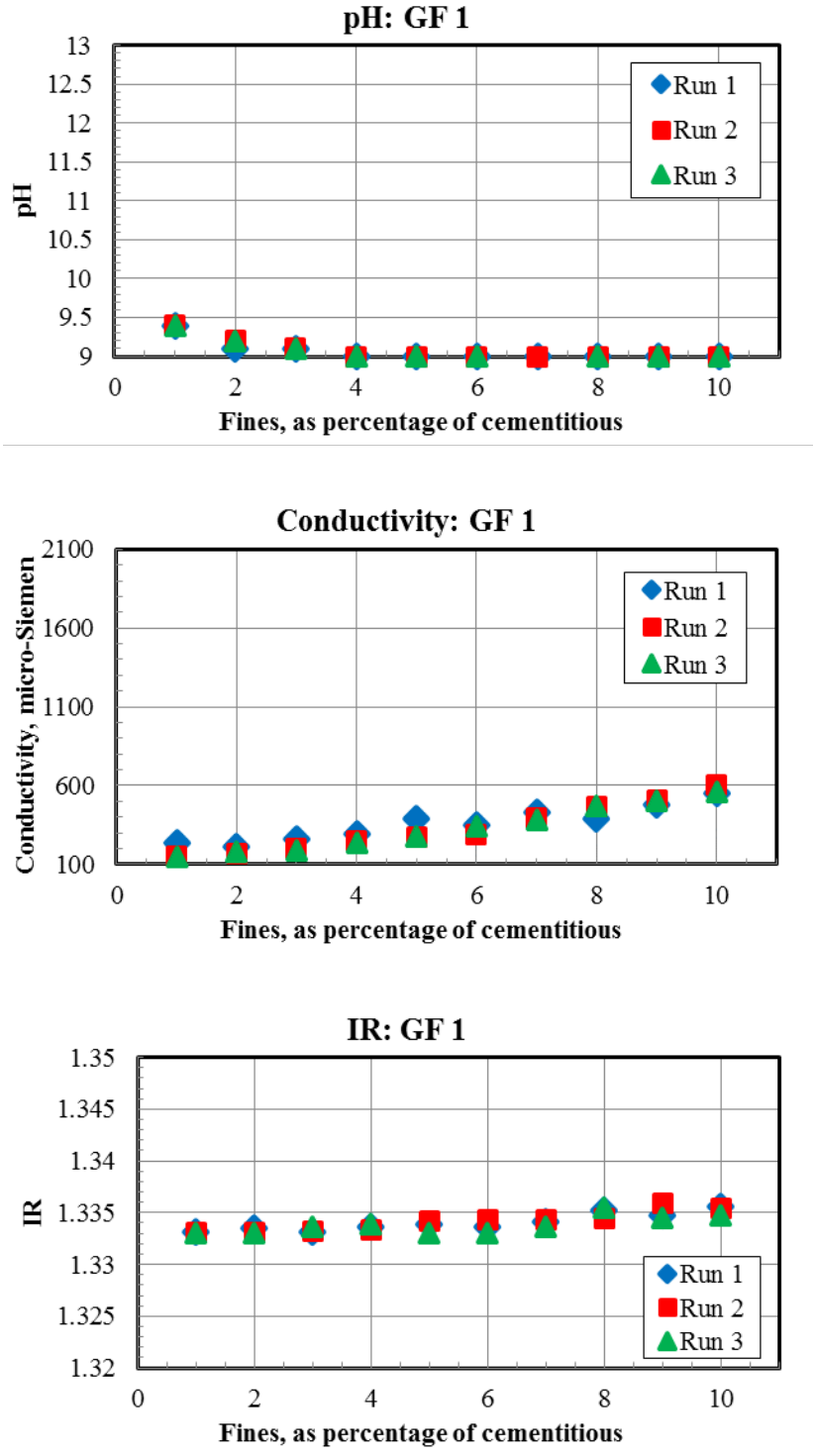


Figure 54. Materials characterization parameter plots for GF 1.

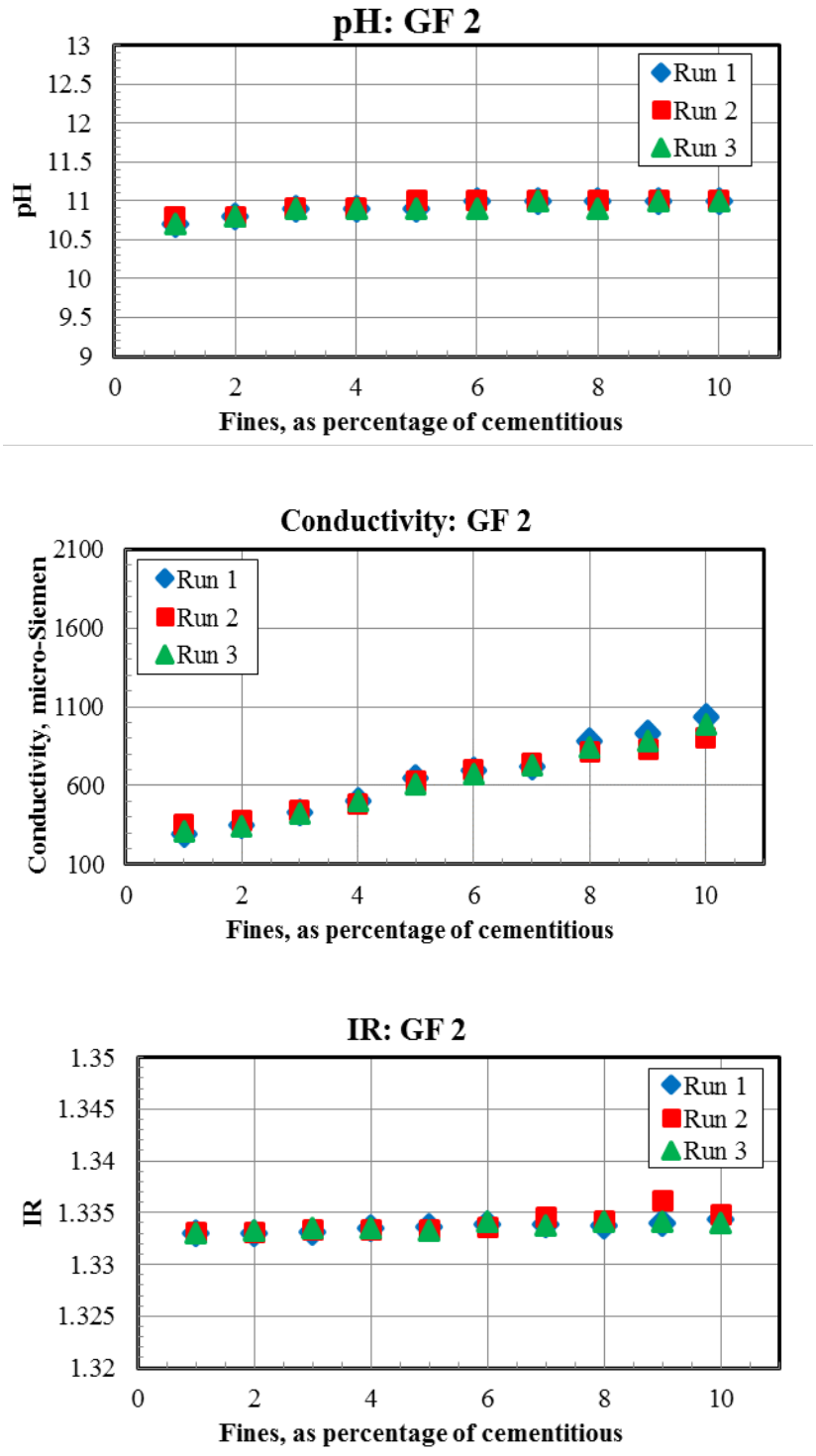
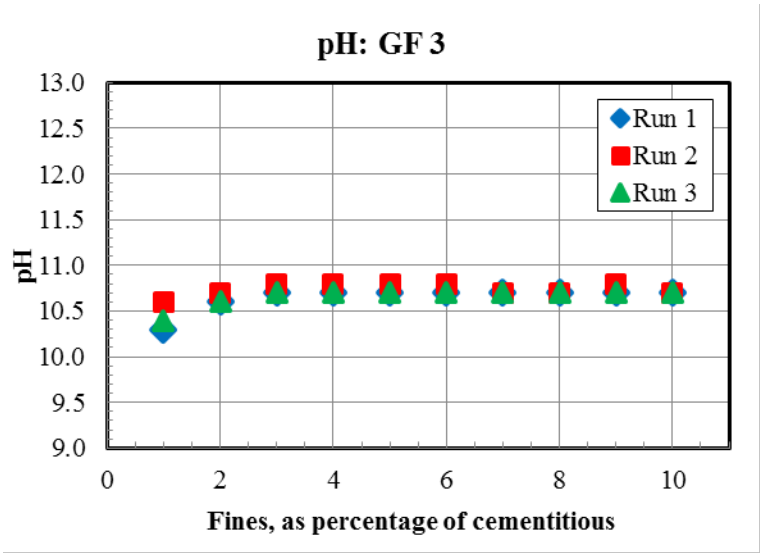
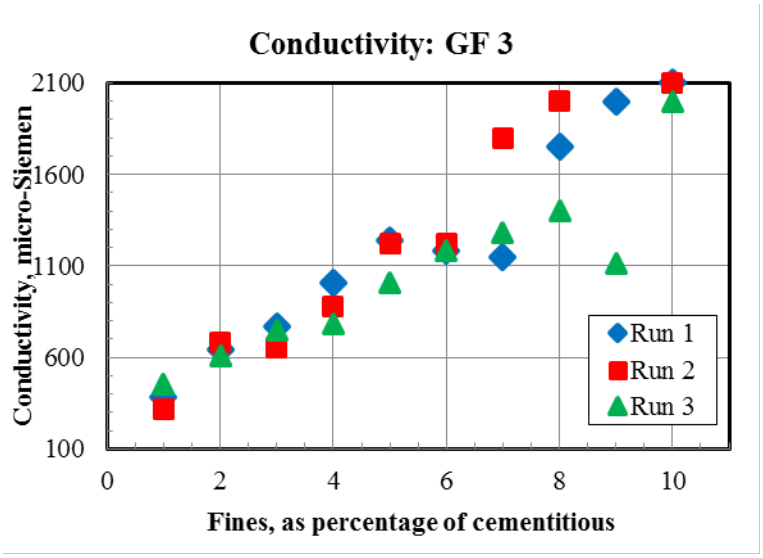


Figure 55. Materials characterization parameter plots for GF 2.





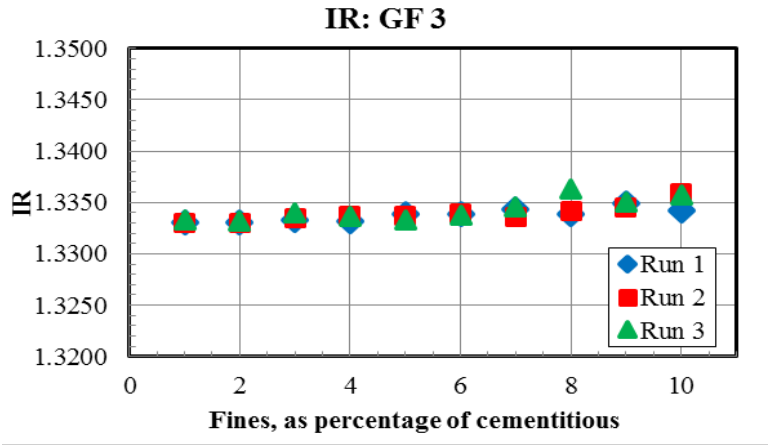


Figure 56. Materials characterization parameter plots for GF 3.

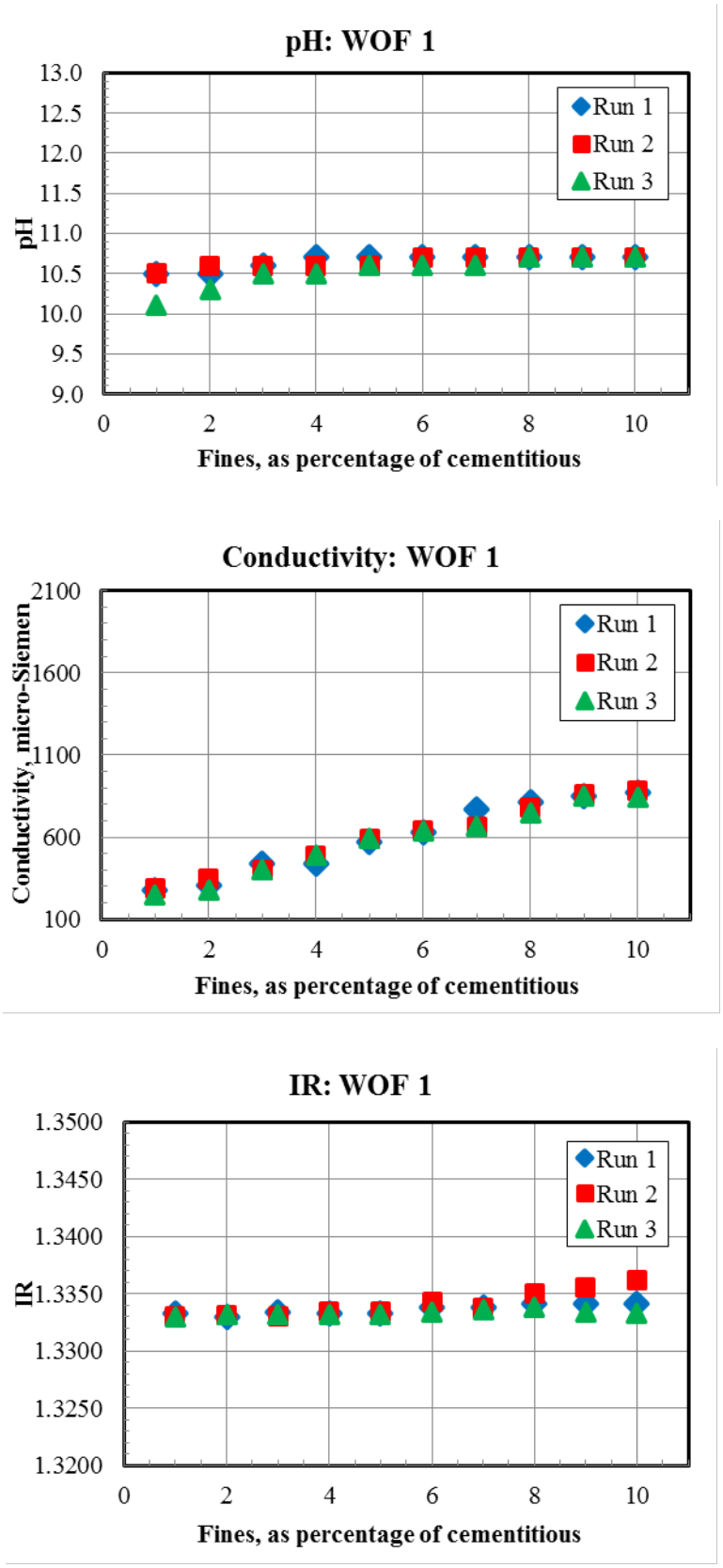


Figure 57. Materials characterization parameter plots for WOF 1.

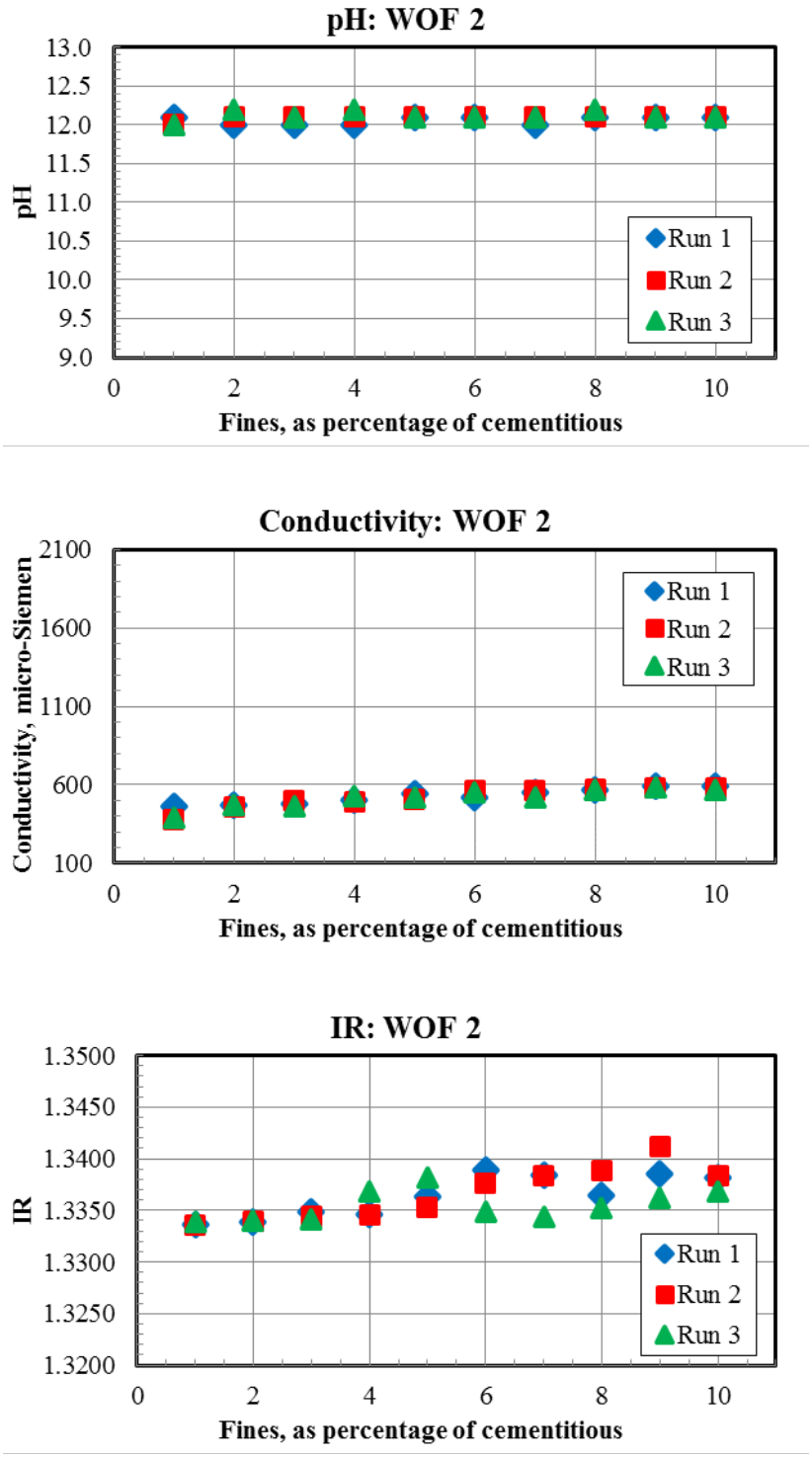


Figure 58. Materials characterization parameter plots for WOF 2.

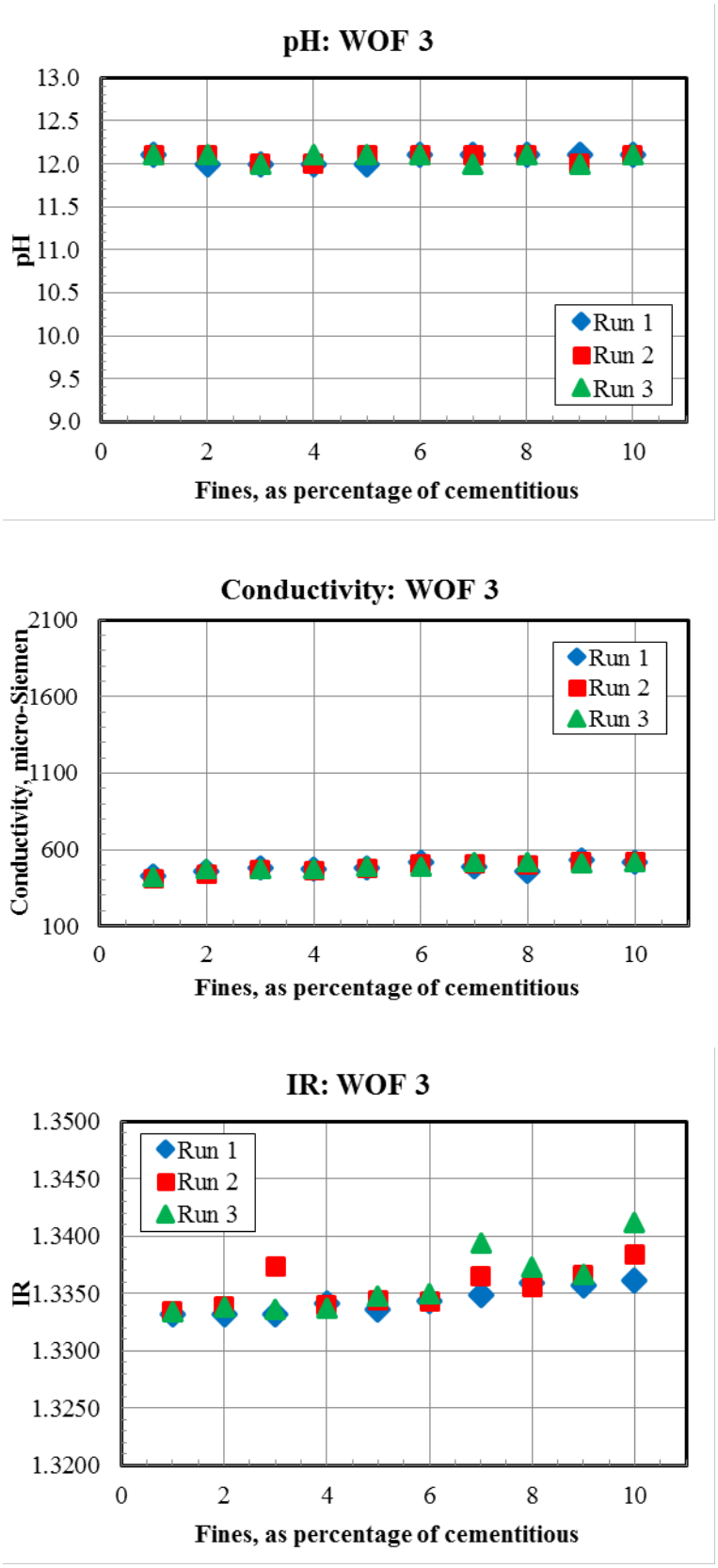


Figure 59. Materials characterization parameter plots for WOF 3.

APPENDIX C

COMPLETE LABORATORY MORTAR TESTING DATA

Table 20. Measured Laboratory Data from Mortar Testing.

Supplementary cementitious material type	Recycled fines source	Percentage of fines as replacement for cementitious materials	3-day compressive strength, psi	28-day compressive strength, psi	Difference in set time, minutes
50% Slag	GF 1	0	2,230	7,310	0
50% Slag	GF 1	2.5	2,410	7,880	-12
50% Slag	GF 1	5	2,400	7,480	-15
50% Slag	GF 1	7.5	2,410	7,180	-35
50% Slag	GF 2	0	3,120	6,760	0
50% Slag	GF 2	2.5	3,120	7,090	-31
50% Slag	GF 2	5	2,910	7,160	-53
50% Slag	GF 2	7.5	2,650	6,720	-65
50% Slag	GF 3	0	2,680	7,380	0
50% Slag	GF 3	2.5	2,650	7,210	-12
50% Slag	GF 3	5	2,340	6,970	-20
50% Slag	GF 3	7.5	2,390	6,980	-31
50% Slag	WOF 1	0	2,570	7,390	0
50% Slag	WOF 1	2.5	2,440	6,940	-17
50% Slag	WOF 1	5	2,670	7,050	-28
50% Slag	WOF 1	7.5	2,560	7,110	-20
50% Slag	WOF 2	0	2,760	7,590	0
50% Slag	WOF 2	2.5	2,720	7,780	-15
50% Slag	WOF 2	5	2,550	7,160	-48

Table 20 (continued)

50% Slag	WOF 2	7.5	2,420	6,740	-109
50% Slag	WOF 3	0	2,330	6,800	0
50% Slag	WOF 3	2.5	2,120	6,580	-1
50% Slag	WOF 3	5	1,910	6,450	-1
50% Slag	WOF 3	7.5	2,040	6,430	-7
37.5% Slag	GF 1	0	3,550	7,370	0
37.5% Slag	GF 1	2.5	3,900	7,400	-39
37.5% Slag	GF 1	5	3,610	7,840	-14
37.5% Slag	GF 1	7.5	4,030	7,750	-32
37.5% Slag	GF 2	0	2,850	7,535	0
37.5% Slag	GF 2	2.5	3,060	7,500	-14
37.5% Slag	GF 2	5	3,260	7,150	-25
37.5% Slag	GF 2	7.5	3,310	7,300	-32
37.5% Slag	GF 3	0	3,310	7,460	0
37.5% Slag	GF 3	2.5	4,460	6,170	-36
37.5% Slag	GF 3	5	4,230	6,520	-42
37.5% Slag	GF 3	7.5	3,790	6,250	-44
37.5% Slag	WOF 1	0	3,080	7,500	0
37.5% Slag	WOF 1	2.5	3,370	7,750	-26
37.5% Slag	WOF 1	5	3,600	7,920	-47
37.5% Slag	WOF 1	7.5	3,360	7,510	-68
37.5% Slag	WOF 2	0	3,330	7,410	0

Table 20 (continued)

37.5% Slag	WOF 2	2.5	3,100	7,450	-73
37.5% Slag	WOF 2	5	3,080	7,210	-102
37.5% Slag	WOF 2	7.5	2,900	6,770	-132
37.5% Slag	WOF 3	0	2,708	7,020	0
37.5% Slag	WOF 3	2.5	2,830	7,040	-13
37.5% Slag	WOF 3	5	3,100	6,980	-58
37.5% Slag	WOF 3	7.5	3,090	6,850	-50
25% Slag	GF 1	0	3,740	7,740	0
25% Slag	GF 1	2.5	4,150	8,270	-16
25% Slag	GF 1	5	4,040	8,350	-33
25% Slag	GF 1	7.5	3,810	7,540	-49
25% Slag	GF 2	0	4,810	8,340	0
25% Slag	GF 2	2.5	4,310	7,200	-4
25% Slag	GF 2	5	4,280	7,020	-37
25% Slag	GF 2	7.5	4,090	6,950	-37
25% Slag	GF 3	0	4,150	7,230	0
25% Slag	GF 3	2.5	4,020	7,030	-16
25% Slag	GF 3	5	4,160	7,140	-26
25% Slag	GF 3	7.5	4,120	6,660	-59
25% Slag	WOF 1	0	4,780	8,230	0
25% Slag	WOF 1	2.5	5,140	8,100	-20
25% Slag	WOF 1	5	4,930	8,180	-19

Table 20 (continued)

25% Slag	WOF 1	7.5	4,690	7,920	-49
25% Slag	WOF 2	0	2,390	6,950	0
25% Slag	WOF 2	2.5	3,000	7,170	-38
25% Slag	WOF 2	5	2,700	6,920	-62
25% Slag	WOF 2	7.5	2,620	6,420	-88
25% Slag	WOF 3	0	3,320	7,250	0
25% Slag	WOF 3	2.5	3,310	7,100	-23
25% Slag	WOF 3	5	3,010	7,100	-40
25% Slag	WOF 3	7.5	2,920	7,110	-62
Control	GF 1	0	3,960	7,350	0
Control	GF 1	2.5	4,560	8,170	-20
Control	GF 1	5	4,860	7,960	-36
Control	GF 1	7.5	4,770	7,910	-56
Control	GF 2	0	4,510	6,590	0
Control	GF 2	2.5	4,680	6,230	-16
Control	GF 2	5	4,610	6,550	-32
Control	GF 2	7.5	4,460	6,410	-38
Control	GF 3	0	4,980	7,040	0
Control	GF 3	2.5	4,940	7,220	-18
Control	GF 3	5	4,940	6,880	-28
Control	GF 3	7.5	4,650	6,940	-31
Control	WOF 1	0	4,510	6,590	0

Table 20 (continued)					
Control	WOF 1	2.5	5,210	6,680	-8
Control	WOF 1	5	4,990	6,840	-24
Control	WOF 1	7.5	4,920	7,180	-42
Control	WOF 2	0	4,510	6,540	0
Control	WOF 2	2.5	4,690	6,540	-27
Control	WOF 2	5	4,590	6,490	-65
Control	WOF 2	7.5	4,340	6,430	-83
Control	WOF 3	0	4,260	7,460	0
Control	WOF 3	2.5	4,440	7,720	-23
Control	WOF 3	5	4,480	7,580	-36
Control	WOF 3	7.5	4,550	7,620	-53
30% Fly Ash	GF 1	0	2,900	5,210	0
30% Fly Ash	GF 1	2.5	2,850	5,430	-30
30% Fly Ash	GF 1	5	2,530	4,960	-23
30% Fly Ash	GF 1	7.5	2,510	5,040	-53
30% Fly Ash	GF 2	0	2,580	5,290	0
30% Fly Ash	GF 2	2.5	2,730	5,450	5
30% Fly Ash	GF 2	5	3,120	5,130	-32
30% Fly Ash	GF 2	7.5	2,900	4,900	-53
30% Fly Ash	GF 3	0	3,380	5,620	0
30% Fly Ash	GF 3	2.5	3,410	5,580	-21
30% Fly Ash	GF 3	5	3,360	5,330	-31

Table 20 (continued)

30% Fly Ash	GF 3	7.5	3,190	5,290	-53
30% Fly Ash	WOF 1	0	3,330	6,010	0
30% Fly Ash	WOF 1	2.5	3,280	6,040	-42
30% Fly Ash	WOF 1	5	3,330	6,040	-66
30% Fly Ash	WOF 1	7.5	3,160	5,980	-100
30% Fly Ash	WOF 2	0	3,040	5,970	0
30% Fly Ash	WOF 2	2.5	3,390	6,110	-49
30% Fly Ash	WOF 2	5	3,060	5,750	-73
30% Fly Ash	WOF 2	7.5	3,170	5,680	-136
30% Fly Ash	WOF 3	0	2,550	5,020	0
30% Fly Ash	WOF 3	2.5	2,600	5,310	-33
30% Fly Ash	WOF 3	5	2,310	4,890	-64
30% Fly Ash	WOF 3	7.5	2,240	4,640	-88
20% Fly Ash	GF 1	0	3,960	6,330	0
20% Fly Ash	GF 1	2.5	3,820	6,010	-28
20% Fly Ash	GF 1	5	3,750	6,560	-36
20% Fly Ash	GF 1	7.5	3,160	5,950	-23
20% Fly Ash	GF 2	0	3,350	6,360	0
20% Fly Ash	GF 2	2.5	3,510	6,200	-45
20% Fly Ash	GF 2	5	3,230	6,240	-60
20% Fly Ash	GF 2	7.5	3,790	5,750	-90
20% Fly Ash	GF 3	0	4,130	6,600	0

Table 20 (continued)

20% Fly Ash	GF 3	2.5	4,280	6,900	-13
20% Fly Ash	GF 3	5	3,860	6,070	-20
20% Fly Ash	GF 3	7.5	3,910	6,140	-34
20% Fly Ash	WOF 1	0	2,990	6,790	0
20% Fly Ash	WOF 1	2.5	2,880	7,040	-21
20% Fly Ash	WOF 1	5	2,920	6,800	-56
20% Fly Ash	WOF 1	7.5	3,230	6,750	-93
20% Fly Ash	WOF 2	0	4,280	6,560	0
20% Fly Ash	WOF 2	2.5	4,020	6,520	-37
20% Fly Ash	WOF 2	5	4,180	6,570	-75
20% Fly Ash	WOF 2	7.5	3,790	6,430	-96
20% Fly Ash	WOF 3	0	3,130	5,910	0
20% Fly Ash	WOF 3	2.5	2,700	5,960	-11
20% Fly Ash	WOF 3	5	2,130	5,520	-9
20% Fly Ash	WOF 3	7.5	2,670	5,620	-19
10% Fly Ash	GF 1	0	4,420	7,300	0
10% Fly Ash	GF 1	2.5	4,660	7,790	-18
10% Fly Ash	GF 1	5	4,450	7,480	-21
10% Fly Ash	GF 1	7.5	4,500	7,340	-32
10% Fly Ash	GF 2	0	4,460	6,300	0
10% Fly Ash	GF 2	2.5	4,270	6,020	-22
10% Fly Ash	GF 2	5	4,200	6,170	-38

Table 20 (continued)

10% Fly Ash	GF 2	7.5	4,090	6,170	-49
10% Fly Ash	GF 3	0	4,100	6,680	0
10% Fly Ash	GF 3	2.5	4,080	6,610	-38
10% Fly Ash	GF 3	5	3,790	6,300	-27
10% Fly Ash	GF 3	7.5	3,840	6,430	-33
10% Fly Ash	WOF 1	0	4,060	6,170	0
10% Fly Ash	WOF 1	2.5	4,460	6,380	-11
10% Fly Ash	WOF 1	5	4,300	6,410	-31
10% Fly Ash	WOF 1	7.5	4,180	6,230	-60
10% Fly Ash	WOF 2	0	4,050	6,400	0
10% Fly Ash	WOF 2	2.5	4,300	6,560	-29
10% Fly Ash	WOF 2	5	3,740	6,270	-49
10% Fly Ash	WOF 2	7.5	3,490	5,670	-76
10% Fly Ash	WOF 3	0	3,600	6,730	0
10% Fly Ash	WOF 3	2.5	3,570	6,370	-15
10% Fly Ash	WOF 3	5	3,480	6,390	-48
10% Fly Ash	WOF 3	7.5	3,240	6,230	-23

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